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# TITANIUM DIBORIDE ELECTRODEPOSITED COATINGS

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MATERIALS SCIENCES DIVISION

June 1977

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**SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)**

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM	
1. REPORT NUMBER AMMRC TR 77-17		3. RECIPIENT'S CATALOG NUMBER	
4. TITLE (and Subtitle) TITANIUM DIBORIDE ELECTRODEPOSITED COATINGS		5. TYPE OF REPORT & PERIOD COVERED Final Report	
7. AUTHOR(s) Jordan D. Kellner,* William J. Croft, and Lawrence A. Shepard		6. PERFORMING ORG. REPORT NUMBER	
9. PERFORMING ORGANIZATION NAME AND ADDRESS Army Materials and Mechanics Research Center Watertown, Massachusetts 02172 DRXMR-D		8. CONTRACT OR GRANT NUMBER(s)	
11. CONTROLLING OFFICE NAME AND ADDRESS U. S. Army Materiel Development and Readiness Command, Alexandria, Virginia 22333		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS D/A Project: 1T162102AH84 AMCMS Code: 612105.H8400 Agency Accession: DA OD 4768	
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE June 1977	
		13. NUMBER OF PAGES 46	
		15. SECURITY CLASS. (of this report) Unclassified	
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE	
16. DISTRIBUTION STATEMENT (of this Report)  Approved for public release; distribution unlimited.			
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)			
18. SUPPLEMENTARY NOTES  *United Technologies Research Center, East Hartford, Connecticut 06108			
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)  Coatings Abrasion resist coatings Titanium boride Erosion resist coatings			
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)			

(SEE REVERSE SIDE)

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE(When Data Entered)

Block No. 20

ABSTRACT

A method of electrodepositing titanium diboride from a low temperature fused salt bath is described. Several applications including the coating of tools are presented. Tests have been run on these tools. A statistical analysis of this data shows a significant increase in tool life in drills, inserts, and end mills when coated with 0.3 mil of  $TiB_2$ , especially when used on fiberglass workpieces.

Cost comparisons for selected production machining operations are presented and show that significant overall savings, including tool costs and labor costs for setup and operation, can be realized through the use of tools coated with electrodeposited  $TiB_2$ .

A laser protection application and an erosion protection application are described.

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## PREFACE

This work was done as a cooperative effort between AMMRC and UTRC. The original plating experiments and the plating process were developed by Dr. Kellner at UTRC. The scientific study was carried out cooperatively by the two groups. All of the tool coating was done at UTRC under a PEMA program, and the tool testing at both AMMRC and UTRC. The statistical analysis of tool life data was performed at UTRC. This final report on the program was prepared jointly.

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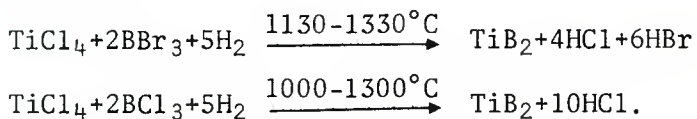
## I. INTRODUCTION

The protection of metallic surfaces against erosion and chemical attack is a continuing problem both to the Army and to the aircraft industry. There has been interest over a period of several years in using boron or various borides as a protecting medium. The reason for selecting boron and the borides for this purpose are their possession of an unusual combination of properties including high hardness, low to moderate density and resistance to chemical attack, good adherence, and a low coefficient of expansion. There are a number of ways that boron and borides could be deposited onto a metallic surface. The method used in this work is electrodeposition from a fused salt. The original work on this project was begun at the Hamilton Standard Division of United Aircraft in 1970 on the deposition of boron.<sup>1</sup> At the same time there was an interest in boron as a protective coating at AMMRC.<sup>2</sup> The electrodeposition process for boron is extremely sensitive to small amounts of contamination of the electrolyte caused by air or moisture. One technique that was tried to alleviate this contamination was to introduce titanium into the electrolyte to remove the oxide contamination. The use of titanium in the electrolyte resulted in the detection of some titanium diboride along with the boron in the deposit. The further refinement of this technique eventually resulted in the electrodeposition of pure titanium diboride. This material seemed superior to boron and led to the program described in this report. Examples of tools coated by this process are shown in Figure 1. In addition to erosion resistance, this investigation has looked into the value of titanium diboride coatings to increase the life of a variety of machine tool cutters. An analysis of the data collected in evaluating these tools is presented. Some of the studies and evaluations were done under AMMRC sponsorship at United Technologies Research Center.

### 1. Chemistry

There are a variety of ways in which titanium diboride can be prepared.<sup>3,4</sup> Direct synthesis from the elements is mainly of interest for fundamental research. It can also be prepared by reacting titanium hydride and boron. It is possible to reduce the titanium oxide with boron or a mixture of boron and carbon or boron carbide. These are the borothermic processes. They generally proceed in vacuum above 1000 C and give an impure product.

It has been prepared by co-reduction with hydrogen from halides of titanium and of boron:



This is the van Arkel method and has been used for vapor plating.

1. KELLNER, J. D. *Electrodeposition of Coherent Boron*. J. Electrochem. Soc., v. 120, 1973, p. 713-716. Patent No. 3,843,497, (S. Russell et al.).
2. CROFT, W. J., TOMBS, N. C., and FITZGERALD, J. F. *Preparation and Characterization of Boron Films from Diborane*. Mat. Res. Bull., v. 5, 1970, p. 489-494.
3. THOMPSON, R. *The Chemistry of Metal Borides and Related Compounds*. Prog. in Boron Chem., v. 2, 1969, p. 173-230.
4. LUNDSTROM, T. *Preparation and Crystal Chemistry of Some Refractory Borides and Phosphides*. Arkiv for Kemi, v. 31, 1969, p. 227-266.





Legend:

- |                                    |                             |
|------------------------------------|-----------------------------|
| a. Carbon-Steel Drills             | e. Carbide Drill Bit        |
| b. High-Speed Steel Lathe Tool Bit | f. Steel Saw Blade          |
| c. Titanium Alloy Turbine Blade    | g. Steel-End Mill           |
| d. Steel Tube Coated Internally    | h. Carbide Tool Bit Inserts |

Figure 1. Examples of titanium diboride electrodeposited coatings on tools.

19-066-1695/AMC-76

It is also possible to react boron trichloride with titanium metal which forms titanium chlorides and free boron which then combines with the titanium substrate to form the diboride.

When the diboride is required in large quantities, it can be prepared by the carbothermic process:





This takes place at about 2000 C. The product is a clinker of very fine crystals. A variety of this method is the reduction of the oxide with boron carbide which is a somewhat faster reaction.

It is possible to prepare the diboride by a reduction process using an active metal such as sodium reacting with titanium dioxide and boric oxide.

## 2. Electrodeposited $\text{TiB}_2$

There has been interest in the past in the electrolytic method of preparing titanium diboride. Andrieux<sup>5,6</sup> and Powell<sup>7</sup> have described the electrodeposition of fine crystals of powdered  $\text{TiB}_2$  from molten baths such as  $\text{MgO}$ ,  $\text{MgF}_2$ ,  $2\text{B}_2\text{O}_3$ , and  $1/2 \text{TiO}_2$  or  $2 \text{CaO}$ ,  $\text{CaF}_2$ ,  $2\text{B}_2\text{O}_3$ , and  $1/4 \text{TiO}_2$ . This material was deposited in the form of a porous mass or loose particles which must be leached in water and acid to remove adherent electrolyte.

Mellors and Senderoff<sup>8,9</sup> reported the electrodeposition of coherent coatings of zirconium diboride from a molten bath of  $\text{LiF-KF-K}_2\text{ZrF}_6$  containing  $\text{KBF}_4$  at 800 C which seems to be the first true electroplating of a boride. Schlain et al.<sup>10</sup> report the electrodeposition of coherent titanium diboride from a borate melt at 900 C, and more recently a patent application<sup>11</sup> was filed by Battelle Memorial Institute that describes a technique similar to the one described in the present work.

A patent was awarded in the U. S. for the UTRC process in April 1976.<sup>12</sup>

## II. PROPERTIES OF $\text{TiB}_2$

### 1. Crystallography

The metal borides are generally a group of compounds that exhibit high hardness, good oxidation resistance, and strength retention at high temperature. The metal diborides such as  $\text{TiB}_2$  are generally the most temperature stable of the metal boron compounds and are characterized by a hexagonal structure.  $\text{TiB}_2$  crystallography is simple hexagonal  $\text{AlB}_2$  type, isomorphous with  $\text{ZrB}_2$  with  $a_0 = 3.027\text{\AA}$ ,  $c_0 = 3.231\text{\AA}$ , and  $c/a = 1.07$ .<sup>13</sup>

The electrodeposited  $\text{TiB}_2$  shows a well-defined preferred orientation. This is predominantly with the poles of the 110 planes perpendicular to the deposition surface. In Table 1 the electrodeposited material is compared with hot-pressed

5. ANDRIEUX, J. L. *Recherches sur l'electrolyse des oxydes metalliques dissous dans l'anhydride borique ou dans les borates fondus*. Thesis, Paris, 1929.
6. ANDRIEUX, J. L. *Preparation des poudres metallique par electrolyse ignee*. Revue de Metallurgie, v. 45, 1948, p. 49-59.
7. POWELL, C. F. *Borides in High Temperature Materials and Technology*. I. E. Campbell and E. M. Sherwood, ed., John Wiley & Sons, Inc., 1967, p. 349.
8. MELLORS, G. W., and SENDEROFF, S. *The Electrodeposition of Coherent Deposits of Refractory Metals, III Zirconium*. Journal Electrochem. Soc., v. 113, 1966, p. 60.
9. MELLORS, G. W., and SENDEROFF, S. Canadian Patent 688,546, June 9, 1964.
10. SCHLAIN, D., McCRAWLEY, F. X., and WYCHE, C. *Electrodeposition of Titanium Diboride Coatings*. Journal Electrochem. Soc., v. 116, 1969, p. 1227-1228; U. S. Patent No. 3697,390, 1972 (McCawley et al.).
11. Patent Application 2214633, File Number P2214633.4, Berlin, Germany.
12. U. S. Patent No. 3,880,729, April 29, 1975.
13. SKAAR, E. C., and CROFT, W. J. *Thermal Expansion of  $\text{TiB}_2$* . J. Am. Chem. Soc., v. 56, 1973.

Table 1. CRYSTALLOGRAPHIC ORIENTATION OF TITANIUM DIBORIDE

Indices	Intensity Ratios			
	Hot-Pressed		Electro-deposited	Powder
100/001	8.0/8.0 = 1.0	5.0/2.0 = 2.5	76/5 = 15.2	60/20 = 3
101/001	23.0/8.0 = 2.8	24.5/2.0 = 12.3	28/5 = 5.6	100/20 = 5
110/001	4.8/2.0 = 0.6	8.7/2.0 = 4.35	150/3 = 50.0	19/20 = 1

Table 2. DENSITY OF ELECTROPLATED  $\text{TiB}_2$ 

Density (g/cm <sup>3</sup> )	Plating Temp (°C)	Plating Current Density (ma/cm <sup>2</sup> )	Thickness (mils)
4.49	721	33	5
4.50	704	28	3
4.56	706	28	3
4.53	718	28	3
4.50	716	28	3

ceramic material which shows the poles of the 001 planes parallel to the pressing direction. For comparison the data from the random powder are included.

## 2. Density

The X-ray density of the ceramic material is 4.52 g/cm<sup>3</sup>.<sup>13,14</sup> The density of electroplated  $\text{TiB}_2$  as a function of electrolyte temperature, current density, and plate thickness is shown in Table 2.

These density measurements were determined in a density gradient tube. The tube contained a water solution of thallium formate-thallium malonate 50-50 mole percent mixture with a density ranging from about 5 g/cm<sup>3</sup> at the bottom to about 2 g/cm<sup>3</sup> at the top. The entire tube was enclosed in a glass jacket through which water was circulated to maintain a constant temperature. Once established the gradient persisted for several days. Floats of known density were dropped in the tube and used as standards. Densities ranged from 4.49 to 4.56 g/cm<sup>3</sup> as shown in Table 2, similar to the X-ray values for  $\text{TiB}_2$ , and no differences with plating parameters were noted.

## 3. Thermal Properties

The thermal conductivity of  $\text{TiB}_2$  at room temperature is 15 Btu/hr ft° F and the other thermal properties<sup>14</sup> are given in Table 3.

The coefficient of thermal expansion was given in Reference 13 and is shown in Table 4.

Table 3. THERMAL PROPERTIES OF  $\text{TiB}_2$ 

Melting Point	2900±80°C
Heat Content	3573 cal/mole ( $H_T - H_{293} = 10.39T + 3.54 \times 10^{-3}T^2$ )
Heat Capacity	0.15 B/ft° F ( $C_p = 10.93 + 7.08 \times 10^{-3}T$ cal/ml <sub>K</sub> )
Heat of Vaporization	430 kcal/mole
Heat of Formation	-52 kcal/mole
Activation Energy of Formation (Ti+B)	9.15 kcal/mole

Table 4. COEFFICIENT OF THERMAL EXPANSION OF  $\text{TiB}_2$ 

Temp °C	$\alpha_{a_0}$	$\alpha_{c_0}$
25	$7.30 \times 10^{-6}$	$10.27 \times 10^{-6}$
500	7.28	10.23
1000	7.25	10.20

14. SHAFFER, P. T. B. *Handbook of High Temperature Materials, No. 1*. Materials Index, Plenum Press, 1964.

## 4. Resistance to Oxidation

The oxidation rate of ceramic  $\text{TiB}_2$ <sup>15</sup> is given in Table 5 at temperatures from 450 C to 1200 C. The rate increases at temperatures above 600 C for the first hour, but even at 1000 C, the oxidation rate diminishes with time of exposure, probably due to the formation of a protective borate film.

The oxidation resistance of the electrodeposited coating was measured by determining the weight gain at various temperatures in air. Below 900 C the oxidation rate is very small, as shown in Table 6.

As the table indicates, the scatter is so high that there was no significant variation of oxidation rate over the current density, thickness, or temperature range studied. At temperatures over 900 C, rapid oxidation ensued due to the loss of the protective  $\text{B}_2\text{O}_3$  layer formed on the surface.

Table 5. RESISTANCE TO OXIDATION OF CERAMIC  $\text{TiB}_2$

Temp °C	Oxidation time, hr	Change in Weight	
		mg/cm <sup>2</sup>	mg/cm <sup>2</sup> /hr
450	1.	+0.42	+0.42
500	1.	+0.63	+0.63
550	1.	+0.63	+0.63
600	1.	+1.78	+1.78
700	1.	+2.00	+2.00
800	1.	+7.36	+7.36
900	1.	+20.4	+20.4
1000	1.	+12.0	+12.0
1000	0.8	+6.8	+8.5
1000	2.8	+10.	+3.6
1000	9.3	+19.	+2.1
1000	19.	+25.	+1.3
1000	29.	+20.	+0.7
1000	40.	+24.	+0.6
1000	48.	+28.	+0.58
1000	63.	+29	+0.45
1000	82.5	+32.	+0.39
1000	102.	+30.	+0.29
1000	119.	+29.	+0.24
1000	147.	+29.	+0.19
1000	170.	+31.	+0.18
1100	20.	+26.	+1.3
1200	2.	+10.	+5.
1200	5.	+24.5	+4.9
1200	25.	+38.4	+1.54
1200	50.	+62.0	+1.24
1200	75.	+68.1	+0.91
1200	100.	+73.7	+0.74

Table 6. OXIDATION RATE OF ELECTRODEPOSITED  $\text{TiB}_2$

Plating Current Density (ma/cm <sup>2</sup> )	Thickness (mil)	Plating Temp (°C)	Oxidation Rate (mg/cm <sup>2</sup> /hr)
21	0.9	610	1.12
			1.10
24	0.6	600	1.05
40	0.7	627	1.01
			1.14

## 5. Strength

Strength in bending of a cantilevered beam consisting of 1 mil of  $\text{TiB}_2$  on both sides of a stainless steel foil of 2-mil thickness has been measured. The stress at which the stress-strain curve becomes nonlinear was taken as the breaking stress of the coating, given by:

$$S = 6 M_0 / bh^2$$

where b is the width of the beam, h the thickness, and  $M_0$  is the moment. The value of S was 38,000 psi  $\pm$  2,000 for five different samples.

## 6. Bond Strength

An attempt was made to determine the bond strength of  $\text{TiB}_2$  on steel by pulling samples on a tensile strength apparatus. Three plated steel samples were glued so that one sample was sandwiched between. The glue failed before the  $\text{TiB}_2$ -steel bond at shear loads of 1080 psi to 2184 psi.

15. SAMSONOV, G. V. *Handbook of High Temperature Materials*, No. 2. Properties Index, Plenum Press, 1964.

## 7. Hardness

Microhardness measurements in a Reichert tester at 84 grams indicated a hardness of  $4060 \pm 200$  Vhn. The polished cross section on which this test was run is shown in Figure 2. The  $\text{TiB}_2$  coating on both sides of the 7-mil steel substrate is 3 mils thick. The impression the Vickers diamond made in the steel corresponds to a hardness of 500 Vhn, while the small dots in the coating correspond to the value of 4060 Vhn. For comparison purposes the Vickers hardness numbers of several other compounds follow:

SiC - 2500 Vhn  
TiC - 3200  
 $\text{Al}_2\text{O}_3$  - 3000  
Diamond - 7000

↓

Table 7. EROSION RESISTANCE

Sample	Coating Thickness (mil)	Impingement Angle	
		20°	90°
		Erosion Rate, sec/mil	
TiB <sub>2</sub> on Steel	0.2	5,600-6,060	955-1,130
TiB <sub>2</sub> on Titanium	0.8	6,960-10,720	1,430-2,440
TiC		1,190	
W		16.5	
NiB <sub>2</sub>		40	
Steel		~20	

## 8. Erosion Resistance

Electroplated  $\text{TiB}_2$  has been evaluated for erosion resistance using an S. S. White Airbrasive unit that directs a stream of  $30\mu$   $\text{Al}_2\text{O}_3$  particles with 1000 ft/sec gas velocity at a small area of the sample. The results are listed in Table 7, along with some other coatings for comparison.

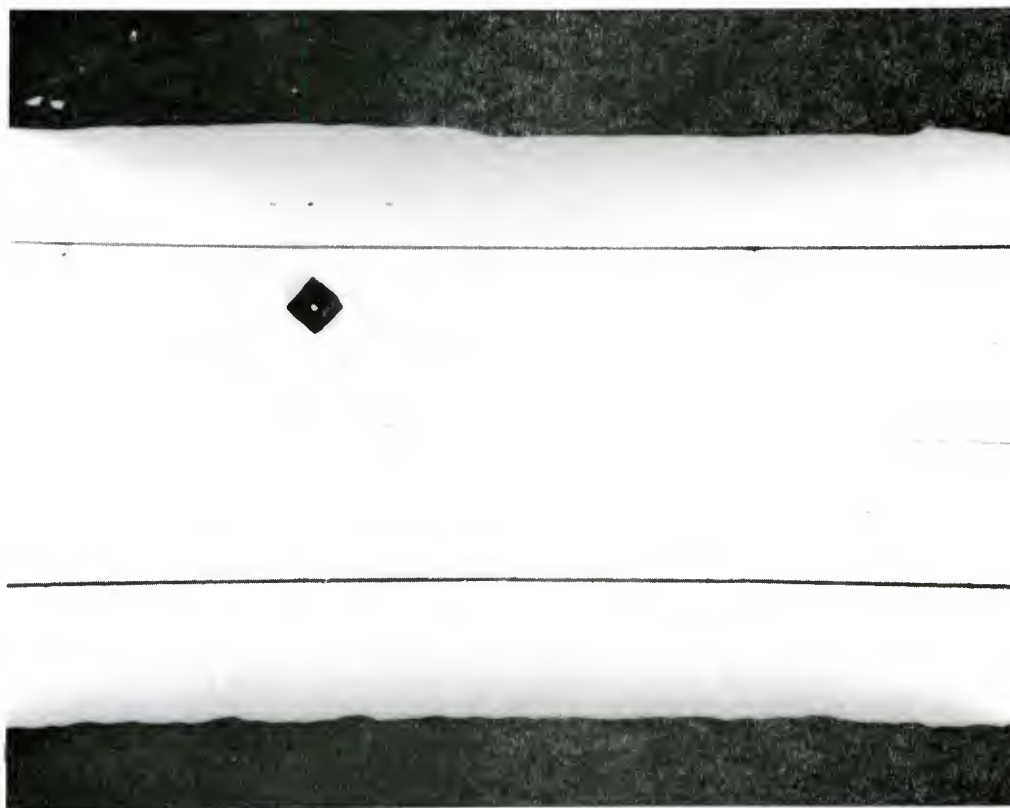


Figure 2. Microhardness of titanium diboride coating,  $4060 \pm 200$  Vhn. Mag. 250X



## 9. Residual Stress

The observations of plated foil specimens have indicated a residual compressive stress is present in the  $\text{TiB}_2$  coating. This stress seems to be proportional to thickness in the thickness range of 0.1 mil to 2 mils. It is particularly evident when the  $\text{TiB}_2$  is deposited on a surface of small radius of curvature, the residual stress causing a partial spalling of the coating. The residual stress is controllable since qualitative experiments indicate it diminishes as current density during plating increases.

### III. PROCESSING

#### 1. UTRC Process for Electrodeposition of $\text{TiB}_2$

A schematic diagram of the plating cell is shown in Figure 3. The electrolyte is a eutectic mixture of the fluorides of potassium, lithium, and sodium<sup>Tw?</sup> melting at 453 C (847 F). The salts are in the weight ratio of 4.64:2.25:1 for  $\text{KF}:\text{LiF}:\text{NaF}$ , respectively. The corresponding mole ratios are 3.3:3.7:1.

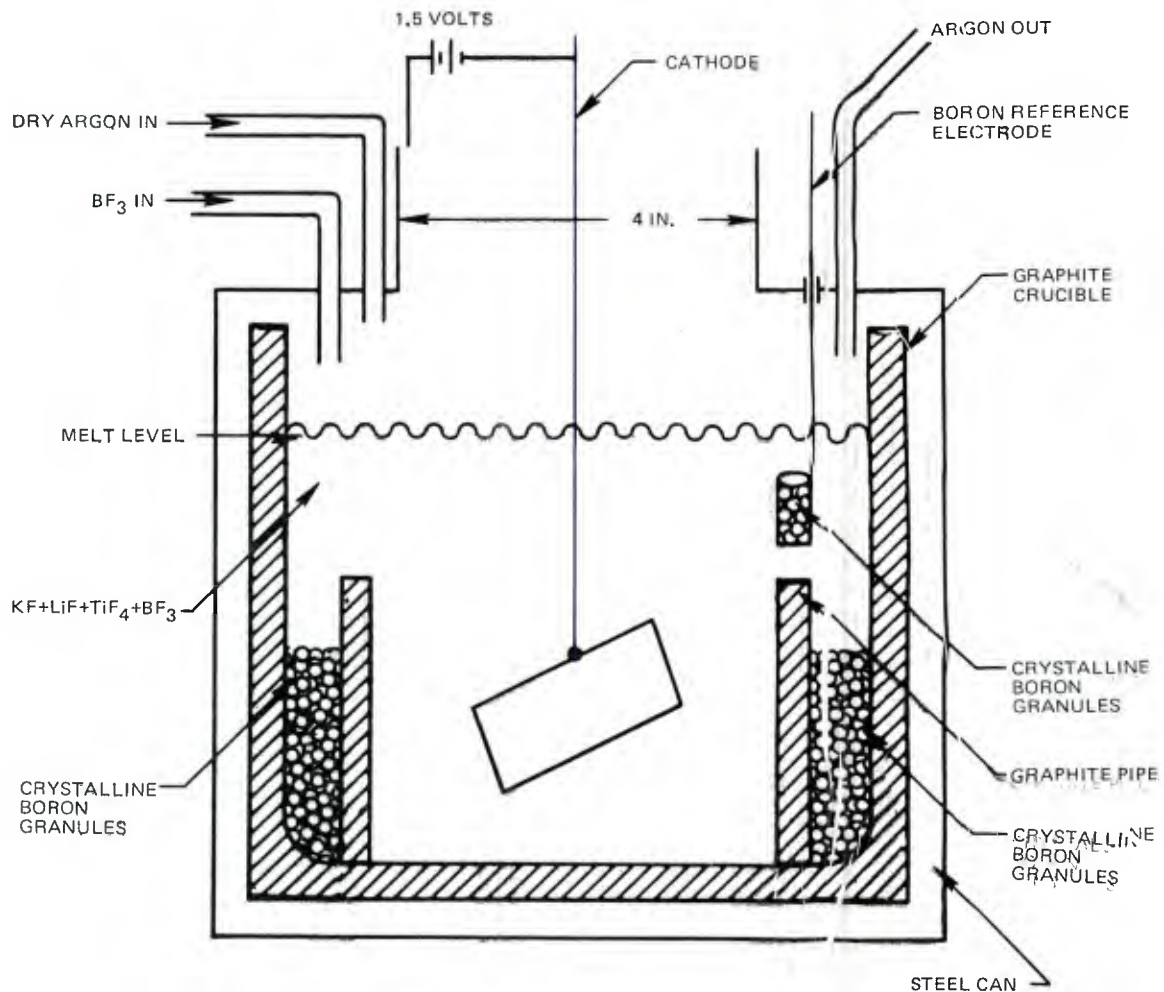


Figure 3. Schematic diagram of plating cell.

The melt is contained in a Union Carbide CS grade graphite crucible enclosed in a 316 stainless steel can. The stainless steel cover is welded and provides welded 316 stainless steel compression fittings for various electrodes to be placed in the melt. Boron nitride inserts are used in the fittings to electrically insulate the electrodes from the can. In laboratory scale units, a 5-inch opening is fitted with a 4-inch aluminum vacuum gate valve in order to provide an argon antechamber. This antechamber is used to prevent contamination of the melt by atmospheric moisture or oxygen. The cover of the antechamber is fitted with Teflon for insulating the cathode from the rest of the cell. A cooling fan directed at the aluminum gate valve prevents the rubber seals from burning. A graphite tube positioned 1/2 inch above the melt surface is used to add  $\text{BF}_3$  gas which dissolves rapidly up to a concentration of about 25 g/100  $\text{cm}^3$ . Flow rates for gas additions up to this concentration are about 100 cu cm/min; thus, about 20 minutes are required for each 25 g of  $\text{BF}_3$ , since 95 percent or more of the gas remains in the melt. The  $\text{BF}_3$  concentration in the melt falls by about 0.5 g  $\text{BF}_3$ /100  $\text{cm}^3$ /day due to the vapor pressure of the gas above the melt, and necessitates the further addition of  $\text{BF}_3$  periodically. The  $\text{BF}_3$  concentration can be monitored by dissolving a one-gram melt sample in water and determining fluoborate ion with an Orion specific ion/tetrafluoborate electrode. The boron added to the melt is 2 to 8 mesh 99.7 percent pure from United Mineral. The titanium is 99.6 percent pure 40-mil wire from the same source. The  $\text{BF}_3$  gas is 99.9 percent pure obtained from Matheson Company in a high pressure cylinder.

The salts are weighed and mechanically mixed in a dry box and placed in the cell. Then the salts are dried under a stream of argon at just under the melting temperature for two weeks.

After the salts are melted and brought to 600 C (1112 F),  $\text{BF}_3$  gas is added to bring the tetrafluoborate ion ( $\text{BF}_4^-$ ) concentration to 5 to 10 g/100  $\text{cm}^3$  and 50 g of boron and 150 g of titanium per kilogram of melt are added and allowed to settle on the bottom of the cell to serve as the anode. Periodic additions of titanium and boron are necessary to keep up the titanium ion concentration (probably  $\text{TiF}_6^{3-}$ ) as  $\text{TiB}_2$  is removed from the cell.

A Hewlett Packard DC power supply is used to establish a constant current between cathode and cell. An integrator using a Kiethly 301 differential operational amplifier is used to determine the charge passed during a run. The titanium concentration in the melt is monitored by a wet chemical method on a melt sample dissolved in acid. The  $\text{BF}_4^-$  concentration is determined by the use of an Orion specific ion electrode for tetrafluoborate ion. At  $\text{BF}_3$  concentrations of over 5 g/100  $\text{cm}^3$ , titanium concentrations of at least 2 percent by weight, and temperatures of 750 C or over, current densities of 300 ma/ $\text{cm}^2$  can be applied. This plating rate will allow an accumulation of coherent  $\text{TiB}_2$  on the surface of 5 mils thickness in one hour. Table 8 summarizes the important parameters.

Table 8. PLATING CONDITIONS FOR  
TWO CONCENTRATION VALUES

Concentration of $\text{BF}_3$ (g/100 $\text{cm}^3$ )	Ti Concentration (% by weight)	Temp (°C)	Current Density (ma/ $\text{cm}^2$ )
2	1	530	10-20
2	1	750	50-150
10	2	530	20-30
10	2	750	150-300



## 2. Chemical Analysis and Coulometry

By weighing samples before and after plating, and comparing this deposited  $\text{TiB}_2$  weight with the amount of charge passed, it has been determined that 9 electrons are exchanged in the deposition of each molecule of  $\text{TiB}_2$  at the cathode. This indicates that the titanium is present in the +3 valence state probably as  $\text{TiF}_6^{\equiv}$ , an ion known to exist in fluoride melts that gives the melt its characteristic purple color. Chemical analysis has shown the samples in weight percent to be 69 percent titanium and 31 percent boron, consistent with a nearly pure compound  $\text{TiB}_2$ . The results of the chemical analysis are shown in Table 9.

Two samples of the coating were exposed in a Norelco Diffractometer using  $\text{CuK}_\alpha$  radiation with a nickel filter. Both samples showed  $\text{TiB}_2$  as a major phase, with some  $\text{TiO}_2$ , probably from melt contamination, as a minor phase. A semiquantitative spectrochemical analysis showed Ti and boron present with a trace of magnesium and silicon. The results of an electron microprobe analysis of early samples containing vanadium (titanium alloy 4911 was used as the anode) are shown in Table 10.

Table 9. COULOMETRIC ANALYSIS

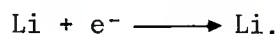
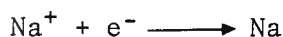
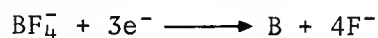
Sample	n Number	Sample Wt (mg)	Ti Wt (mg)	Ti Wt%
7BTB-128	8.9	162	114	70.4
7BTB-128B	8.95	162	111	68.9
7BTB-129	9.24	156	109	69.9
Theory - pure $\text{TiB}_2$	9			68.9

Table 10. ELECTRON MICROPROBE ANALYSIS

	Weight % Titanium + Vanadium	
	Sample 1	Sample 2
Top. of Layer	68.5	70.5
Middle of Layer	68.1	70.2
Base of Layer	67.3	69.4

## 3. Electrode Processes

At the cathode the following reactions may occur:



The last three reactions take place at much higher cell voltages than the first two, and probably make a negligible contribution to the cell current at operating voltages. The boron and titanium produced react on the surface or immediately before deposition to form  $\text{TiB}_2$  with 9 electrons exchanged during the entire process for each molecule of  $\text{TiB}_2$  produced. At the anode the following reactions are possible:



The first reaction requires a high potential and does not occur to any appreciable extent. The next three reactions will occur in a melt that has oxide contamination. This contamination is very difficult to remove since both the oxygen and carbon dioxide produced by these reactions are extremely soluble in the electrolyte. The last two reactions occur at operating voltages in clean melts and replenish the electrolyte concentration of electroactive species that are consumed at the cathode during electrodeposition.

#### 4. Coating Appearance

Figure 4 shows a polished and etched cross section of a 2-mil  $\text{TiB}_2$  coating on steel at 500X. The columnar nature of the crystal grain structure is evident.

Surface roughness appears in deposits from older electrolytes, probably caused by bubble formation from melt contamination by oxygen and moisture from the atmosphere. Other causes are inadequate substrate preparation and particulate matter dispersed in the electrolyte by disintegrating anode granules causing solid bumps by electrostatic attachment to the cathode surface.

New melts that have been carefully dried produce deposits up to 2 mils thick with virtually none of the above surface irregularities and have a surface finish of about 50 microinches rms. At thicknesses over a mil, surface roughness increases. Coating thicknesses of about 100 mils have been grown on tantalum foil, but these have exhibited surface irregularities, defects, and subsurface voids.

#### 5. Rotating Cathode Experiment

The effect of cathode rotation on the  $\text{TiB}_2$  electroplate quality was studied as a function of current density and rotation rate. At current densities ranging

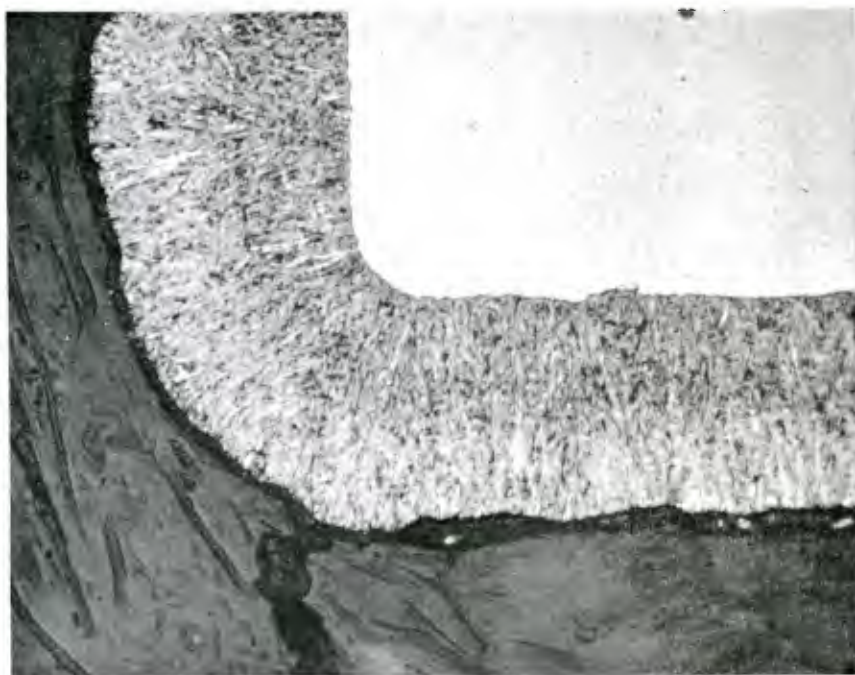


Figure 4. Cross section of titanium diboride coating.

from 20 to 120 ma/cm<sup>2</sup>, rotation at rates from 20 to 100 rpm resulted in plates of lower quality than without rotation. The TiB<sub>2</sub> on the rotated specimens was powdery, of a sooty color and texture and nonadherent. Table 11 is a summary of the current densities, rotation rates, and plate conditions that were found.

#### IV. APPLICATIONS

##### 1. Turbine Engine Blades

The applications considered to date for electroplated TiB<sub>2</sub> are those involving erosion protection or wear protection. The two large categories are steel compressor blades and stators for turbine engines and cutting tools.

Figure 5 is a photograph of a 16-inch steel stator from the compressor section of the Pratt and Whitney engine, the JT-12. This stator was coated with a 0.7-mil TiB<sub>2</sub> deposit for erosion protection and underwent a static engine test for 500 hours with no damage. The fatigue strength of TiB<sub>2</sub>-coated steel compressor blades has been determined to be only 15 to 20 percent less than the value for uncoated blades.

Table 11. EFFECT OF CATHODE ROTATION ON TiB<sub>2</sub> PLATE QUALITY

Rotation Rate (RPM)	Current Density (ma/cm <sup>2</sup> )	Plate Quality
0	75	G
0	50	VG
0	25	VG
20	25	NG
30	50	NG
40	50	NG
60	40	NG
60	100	NG
60	120	NG
100	50	NG

Key: G - (gray, hard)  
 VG - (smooth, light gray, very hard)  
 NG - (black, powdery, nonadherent)

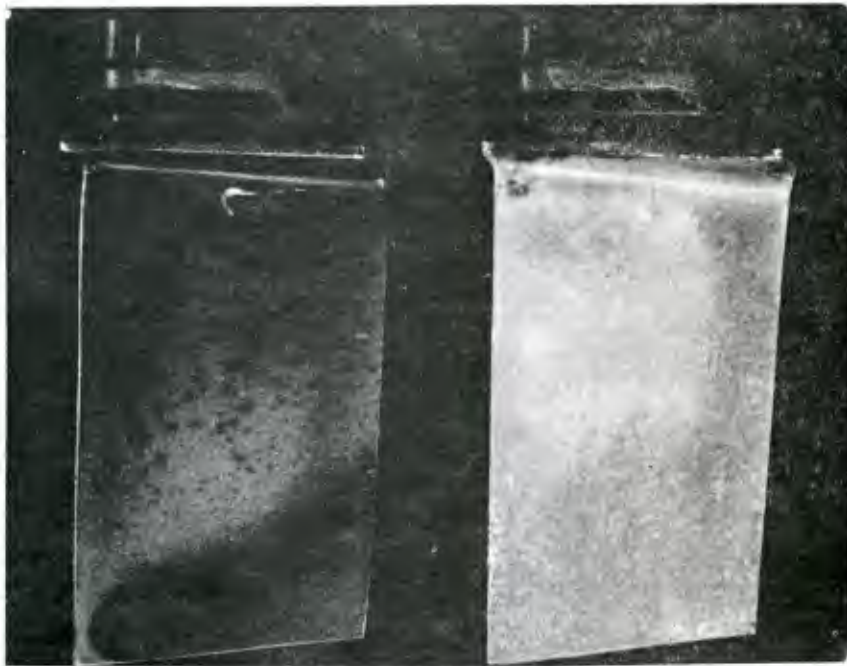


Figure 5. Coated 16-inch steel stator (JT-12 engine).

TiB<sub>2</sub>-coated steel compressor blades of the 10th and 13th stages of the Pratt and Whitney JT-8D are currently undergoing field tests with several airlines. The tests will be successful if the coating can prevent severe trailing edge erosion leading to airfoil shape changes. Figure 6 shows the concave side of an erosion-damaged uncoated 10th stage compressor blade on the left, and a similarly tested undamaged TiB<sub>2</sub>-coated blade on the right.

## 2. Tools

The wear protection applications have centered on machine tools such as steel drills, carbide drills, carbide inserts, and steel end mills. Figure 7 is a photograph of a 1/2" carbide drill coated with TiB<sub>2</sub> to within 3/4" of the top. Figure 8 is a 200X scanning electron micrograph of the tip of a steel drill showing the original surface and the coated surface. This tool and others were tested at UTRC on fiberglass and 4340 steel workpieces. The results of these tests are very encouraging, and will be presented later in this report.



a. Uncoated

b. Coated

Figure 6. Demonstration of erosion resistance of titanium diboride coating on vane. Mag. 1X



Figure 7. Carbide drill coated with titanium diboride. Mag. 3X



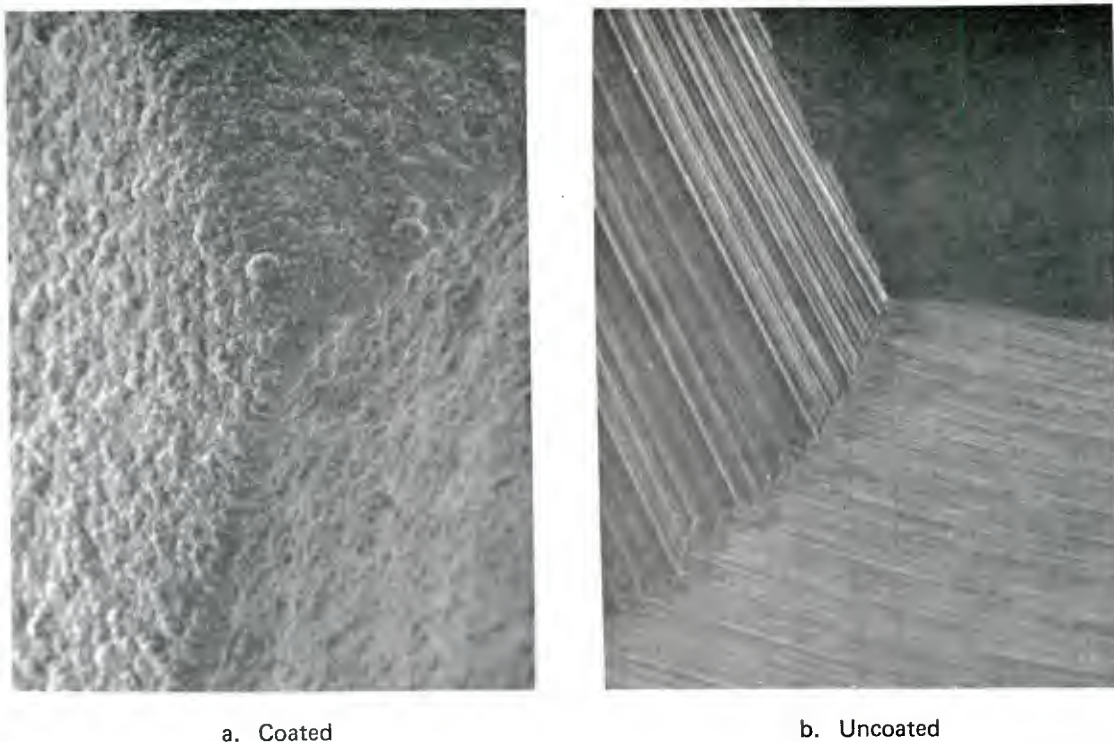


Figure 8. SEM photograph of drill tip before and after coating. Mag. 200X

### 3. Electroformed Bodies

In an attempt to evaluate the possibility of electroforming titanium diboride, a 4" x 4" x 10 mil plate of tantalum was coated with titanium diboride to a thickness of 1/8" on each side. The deposit became porous after an initial thickness of 10 mils was laid down. A section through this specimen is shown as Figure 9.

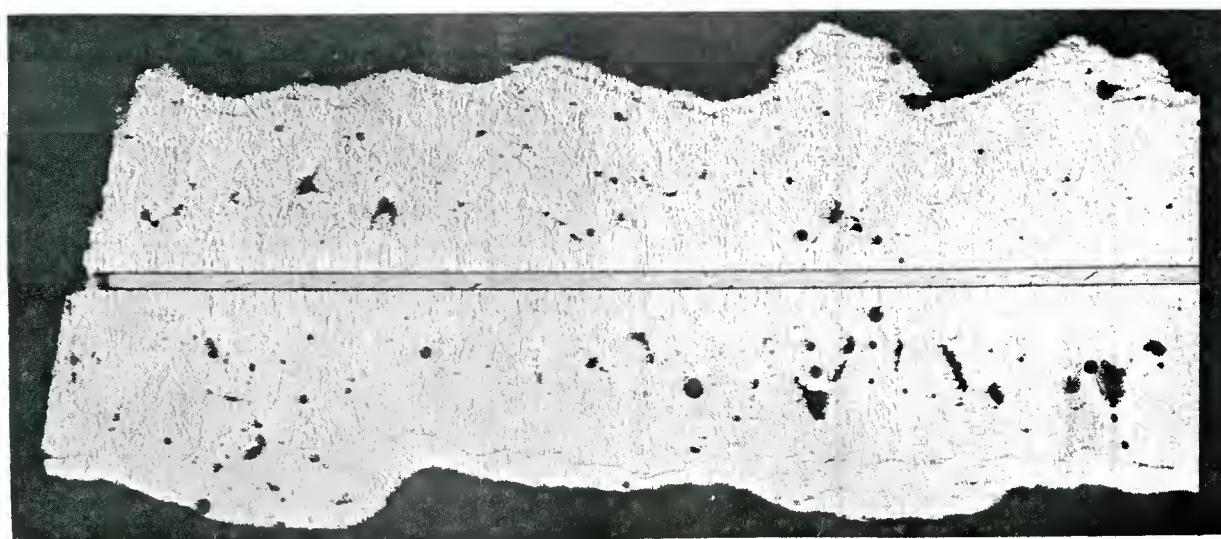


Figure 9. Section of thick electroformed titanium diboride coating. Mag. 15X

#### 4. Laser Protection

Titanium diboride exhibits a number of physical and chemical properties which suggest its potential for a laser protective coating. As noted in Table 12 the compound is very hard and stable, and melts at a high temperature. By contrast, the luster and high electrical and thermal conductivities suggest the material to be almost metallic in character. High reflectivity and rapid heat conduction are certainly desirable properties. The density is advantageously low, and the coefficient of thermal expansion is close to that of steel. In addition, of course,  $TiB_2$  can be electrodeposited at moderate temperatures as a thin, continuous adherent layer upon a metal surface.

Maraging steel was chosen as the substrate material in this test because the optimum aging temperature for maximum hardness of this alloy corresponds to the coating deposition temperature, about 700 C. Thus, aging of the martensite could occur simultaneously with the plating process. The pedigree of the maraging 250 steel disk which was plated is given in Table 13. A two-mil  $TiB_2$  coating was applied by Dr. J. Kellner at United Technologies Laboratory.

The coated sample was irradiated on the AVCO Everett pulsed  $CO_2$  laser with the maximum energy and time available. The laser testing is outlined in Table 14. The available laser energy was considerably less than that required for burnthrough or even cratering in this material. Thus, the subsequent examination of the irradiated coated sample did not produce definitive conclusions regarding the protective effects of the plating. Nevertheless, important evidence was gained in the study.

Table 12.  $TiB_2$  PROPERTIES\*

1. Heat of Formation	= -52 kcal/mole
2. Vickers DPH Hardness	= 3400 kg/mm <sup>2</sup>
3. Melting Point	= 2900 C
4. Linear Coefficient of Expansion (Normal to Surface) <sup>13</sup>	= $7.30 \times 10^{-6}$ /deg C
5. Luster	= Metallic
6. Electrical Resistivity (R.T.)	= 15.2 micro ohm cm
7. Thermal Conductivity	= 0.060 cal/cm deg sec

\*Data from Reference 14.

Table 13. MARAGING STEEL DISK

a. Material - Maraging Grade 250, Type RSM  
Annealed 1/4-Inch Plate

b. Supplier - Republic Steel Corporation

c. Average Chemistry

<u>Element</u>	<u>Weight Percent</u>
Ni	17-19
Co	7-8
Mo	3.5-4.5
Ti	0.36
Al	0.05-0.15
Fe	Remainder

d. Condition - as received; sample was Blanchard  
ground to a 20 rms finish

Table 14. LASER IRRADIATION

a. Sample was subjected to a pulsed $CO_2$ - AVCO, Everett
1. Approximate area of spot = 5 cm <sup>2</sup>
2. Total number of pulses per second = 125
3. Total number of pulses used = 257
4. Energy transferred from laser = 29,300 joules
5. Average intensity = 2.8 kW/cm <sup>2</sup>
6. Peak intensity = 1.5 megawatts/cm
7. Average peak = 5.6 kW/cm <sup>2</sup>
8. Pulse width = 1.5 milliseconds
9. Wave length = 10.6 microns
10. Pulse shape = square
11. Beam profile = gaussian, peaked at the center
b. Reflectivity of $TiB_2$ plated sample at 10.6 microns = 14%



The appearance of the plated disk after irradiation is shown in Figure 10. The 2.5-cm-diameter circle indicated by the arrow encompasses the approximate outline of the laser beam. The faint darkening within the circle represents the only visible damage.

Overall, the  $\text{TiB}_2$ -coated disk showed a blue-grey velvety appearance, with the exception of the spots around the edge which appear dark on the photo, and shiny in reflection. Microscopic examination revealed the velvety area to be the crystalline facets of the plated  $\text{TiB}_2$  layer. The darker spots are regions where the  $\text{TiB}_2$  coating delaminated to about half thickness. Nowhere was the steel substrate found to be exposed, nor did the coating spall off during cutting and polishing.

The sample was diamond sawed along the lines indicated in Figure 11 and sections marked Cr and Cl at the center of and well outside the irradiated area were examined as indicated in Table 15.

The results of these tests may be summarized as follows:

a. Sample Surface

Both the X-ray and nondispersive X-ray analysis results indicated only crystalline  $\text{TiB}_2$  on the sample surface, as indicated above. The substrate was nowhere exposed. No reaction occurred as the result of irradiation.

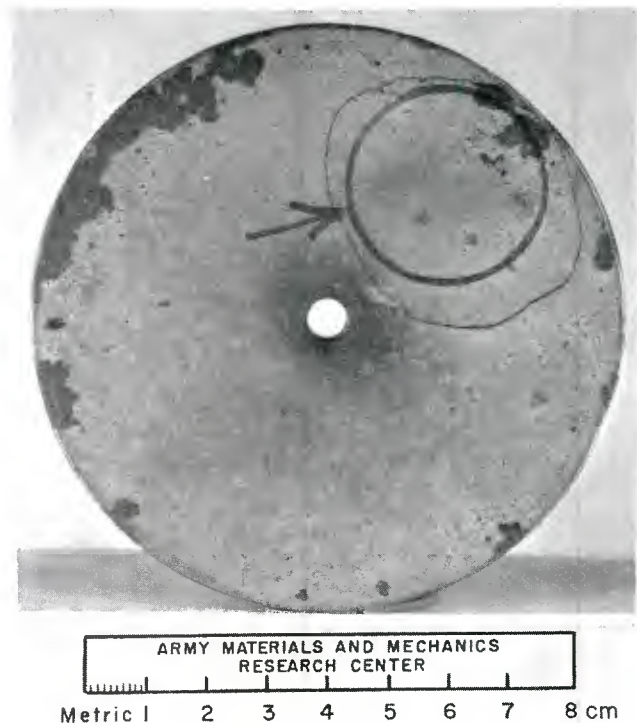


Figure 10. Laser-irradiated titanium diboride coating on maraging steel.

19-066-217/AMC-74

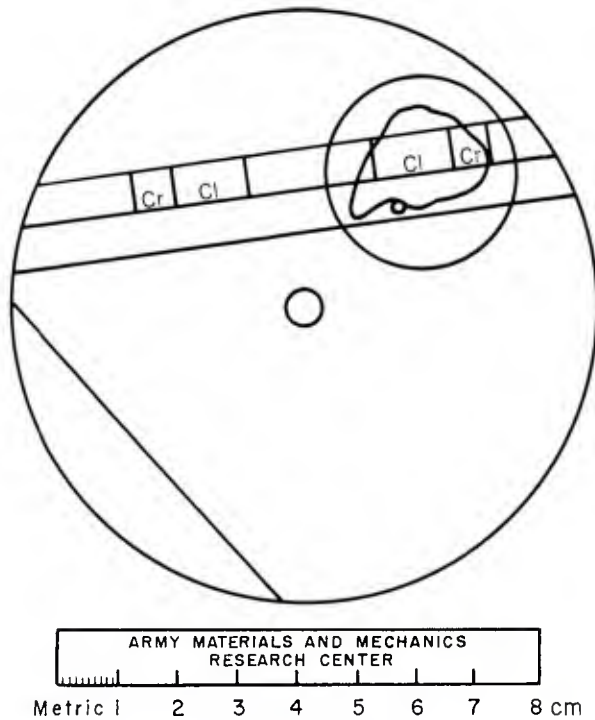


Figure 11. Specimen layout for irradiated maraging steel sample.

Table 15. INVESTIGATION OF LASER-IRRADIATED TiB<sub>2</sub>-PLATED MARAGING STEEL DISK

Research Program. (At each step, both the lased and unlased regions of the sample were examined.)

1. X-ray of Surface
2. Cutting of Disk
3. Sections Marked Cr — Electron Diffraction  
Photographs of Surface
4. Sections Marked Cl

Surface Studies

- a. Scanning Electron Microscope
- b. Light Microscopy
- c. Nondispersive X-ray Analysis

Polished Cross Section

- a. Microstructures
- b. Microhardness

b. Effects of Plating and Irradiation on Steel Substrate

A polished and matched section of TiB<sub>2</sub> coating and steel substrate is shown in Figure 12. The columnar grain structure of the TiB<sub>2</sub> is delineated by polarized light.

Below the micrograph are given microhardness tests results for specimens in the lased and unlased regions. There are essentially no hardness differences between the two regions.

An interesting observation which appears to be significant is the roughly 20% hardness increase in the steel near the plated interface. This increase suggests that there is some boron diffusion into the substrate during plating.

c. Scanning Electron Microscope, Electron Diffraction

The scanning scope and electron diffraction studies of the lased and unlased regions brought out the only distinguishing effect of laser irradiation. Figure 13 shows the small freckles which appear randomly deposited over the crystal facets in the lased area. In the unlased area, the freckles do not appear.

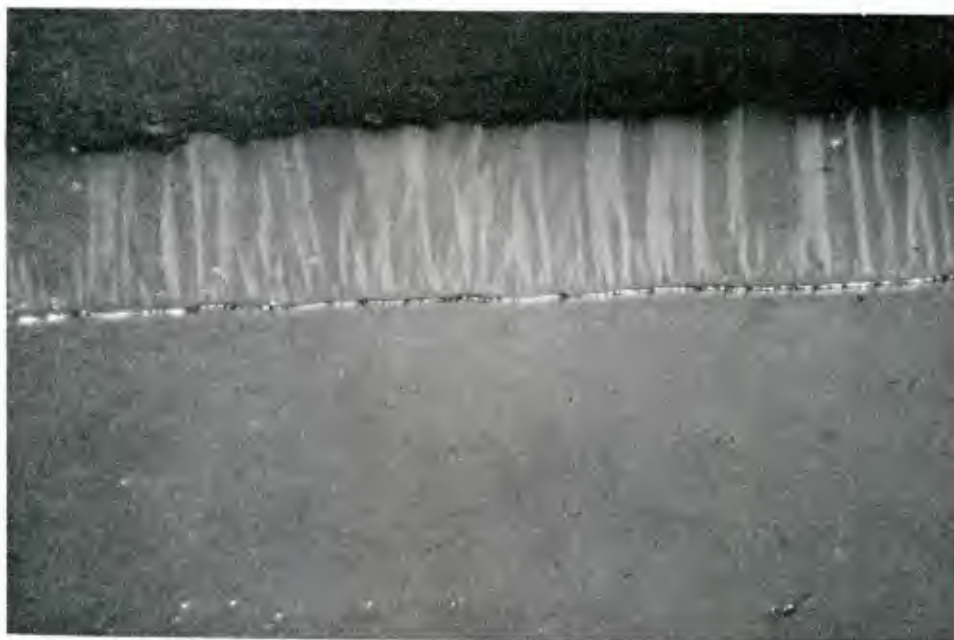


Figure 12. Microhardness of deposited titanium diboride layer.

Material	Lased		Unlased	
	Distance Below Interface, $\mu\text{m}$	Vickers DPH 50-g load	Distance Below Interface, $\mu\text{m}$	Vickers DPH 50-g load
$\text{TiB}_2$	-	5730*?	-	2920
	-	3160*	-	3300*
	-	3810*	-	3720*
Maraging Steel	4.97	480†	8.13	441‡
	11.39	407‡	108	353‡
	14.27	382‡	108	346‡
	467	345‡		

\*Crack at corner of impression

†Gram load<sup>10</sup>

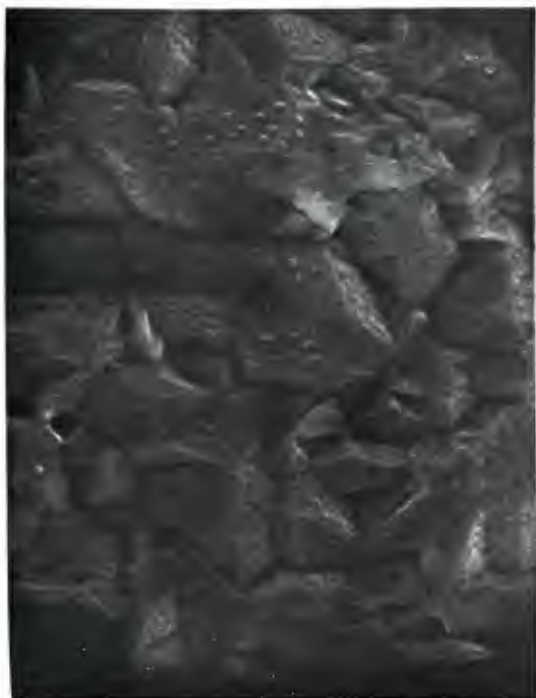
‡Corrected for pincushion effect

Electron diffraction images of these same two areas, Figure 14, show only a single crystal pattern from one large grain in the unlased area; in the lased area, diffraction rings appear, due to the overlay of randomly deposited freckles. The diffraction rings have been identified as  $\text{TiB}_2$  only.

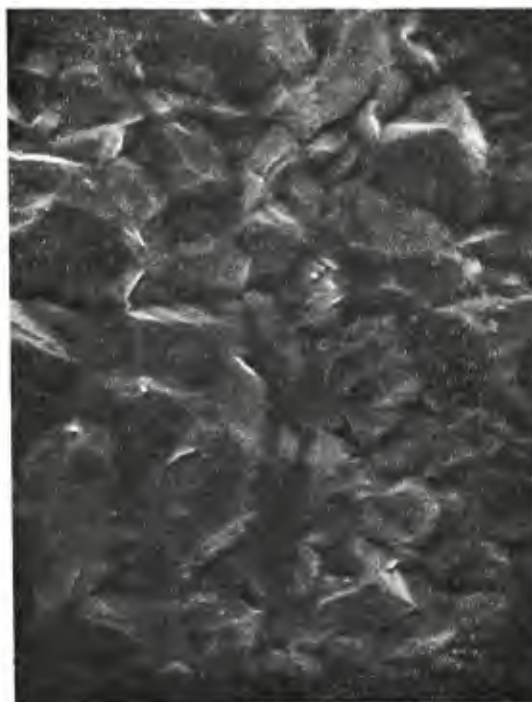
Thus, two important results accrue. First, the  $\text{TiB}_2$  evaporates and redeposits as the stoichiometric compound. Second, there is no evidence of oxidation —  $\text{TiO}_2$  formation — as the result of laser irradiation.

The total energy of laser irradiation used in this test, Table 14, was about one sixth that necessary to burn through the irradiated volume of steel, presuming complete energy absorption. Thus, the effect observed was relatively small, and the efficacy of  $\text{TiB}_2$  coatings in laser protection cannot be established.

The two significant observations, that is a) evaporation and redeposition of stoichiometric  $\text{TiB}_2$  without melting, and b) no oxidation of the  $\text{TiB}_2$  layer, suggest that the coating may offer protection. The low reflectivity of the velvety plated coating, Table 14, suggests that the major portion of the impinging radiation was absorbed.

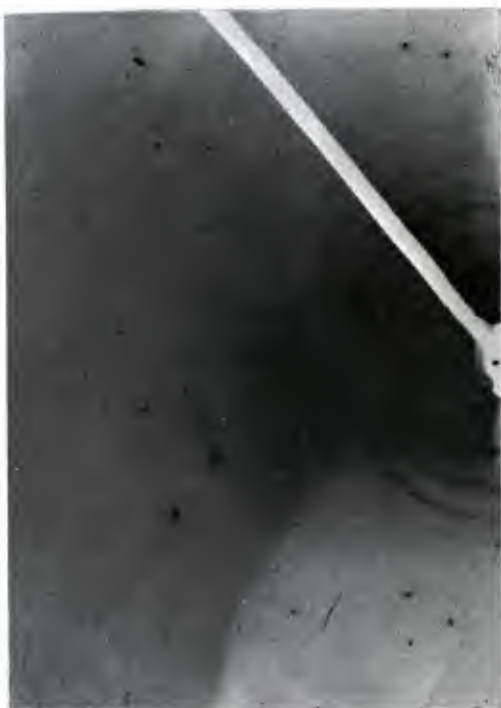


a. Lased Area

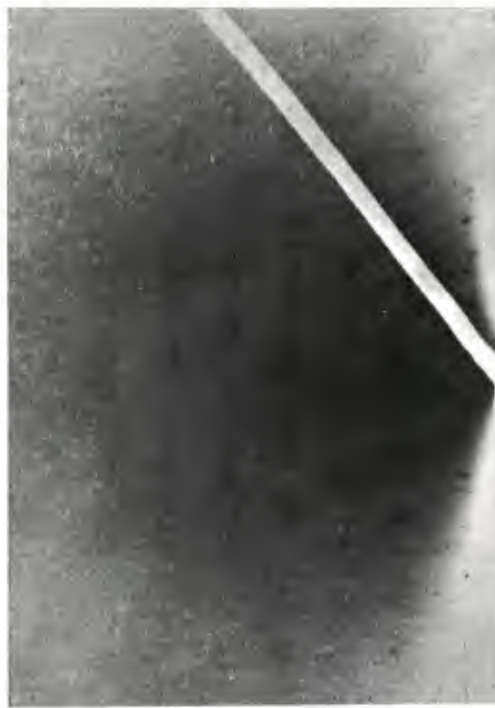


b. Unlased Area

Figure 13. SEM photograph of titanium diboride coating on steel. Mag. 1600X  
19-066-317/AMC-74



a. Lased



b. Unlased

Figure 14. Electron diffraction of titanium diboride-coated steel. Mag. 500X



## 5. Other Applications

Other applications for electrodeposited  $\text{TiB}_2$  include valve stems and seats for erosive environments that exist in coal gasifiers and general wear resistance applications such as journal boxes and bearings.

## V. TOOL TEST PROCEDURE

### 1. Drill Tests on Steel

These drill tests, detailed in Table A-1 in Appendix A, were performed on 4340 steel of two hardnesses, HRC 28 and HRC 42, on two milling machines denoted as Bridgeport 9 and 10. The drills were commercial 1/4-inch, high-speed steel twist drills. Half the 120 drills tested were coated with  $\text{TiB}_2$  to a thickness of 0.3 mil using the procedures outlined in Section I and the Appendix of this report. Table A-1 lists the data in cubic inches removed for several variable conditions such as soft or hard workpiece, coated or uncoated drill, slow or fast feed rate, and no coolant, Lusol coolant, or soluble oil coolant. Each set of conditions was tested at six different speeds that were measured with a stroboscopic tachometer. The table lists the number of holes drilled to 1/2-inch depth for each drill, and the total cubic inches removed for each drill. Both the number of holes drilled and the total material removed have been normalized for the same measured wear on each drill, namely 0.005 inch. The amount of wear produced on each drill was measured under a binocular microscope fitted with a calibrated reticule which enabled the wear to be measured to within one mil. Thus,

$$\text{Normalized number of holes drilled} = \frac{\text{number of holes actually drilled}}{\text{wear land}/0.005''}.$$

In all cases the actual amount of wear varied from 0.002 inch to 0.010 inch.

### 2. Turning Data on Steel

Fifty-six inserts were tested, half of them coated with 0.3 mil of  $\text{TiB}_2$  as outlined in the Appendix. The inserts numbered 1 to 32 and 43 to 56 were 5/8 inch square, grade CY16 tungsten carbide, from Wendt-Sonis/Unimet, style SNMA543F. Those numbered 33 to 42 were a nickel-bonded 1-inch square insert. The turning tests were performed on 4340 steel cylinders of two different hardnesses, HRC 25 (250 Bhn) and HRC 38 (350 Bhn), referred to as soft and hard workpieces. In the first group of tests, numbered 1 to 32 and summarized in Table A-2 there were five 2-level variables, each denoted by the zero level for the lower value and the one level for the higher value.

A quantity called "total material removed" was obtained as follows: each insert was used to remove material from the diameter of the steel cylinder with each cutting edge used for a different spindle speed. The surface speeds ranged from about 80 sfm to 1500 sfm. The wear land was measured microscopically and the material removed was calculated based on a wear land of 0.02 inch. The material removed by all cutting edges totaled together makes up the "total material removed".

Inserts 33 to 42 were tested for a more limited set of conditions as shown in Table A-3. The symbols for the variables have the same definitions as in Table A-2.

The set of tests on cobalt-bonded inserts, numbered 43 to 56, are summarized in Table A-4. The wear rates shown in Table A-4 were obtained using a lead angle of 20°. The data shown in Tables A-2 and A-3 were obtained with the point of the tool perpendicular to the workpiece, a lead angle of zero degrees.

### 3. Drill Tests on Fiberglass

The tests were performed on a G-10 fiberglass sheet 1 inch thick, using 114 1/4-inch, high-speed steel twist drills. Half the drills were coated with 0.3 mil of TiB<sub>2</sub>. Holes were drilled to a depth of 1/2 inch using a Bridgeport Milling Machine with power feed. As in the tests on steel, wear was measured on each drill under the microscope, and the number of holes drilled was adjusted for 0.005 inch of wear. Table A-5 gives the amount of material removed and the number of holes drilled with each drill for a variety of conditions. Drills 37 to 45 and 112 to 114 were tested with center holes drilled before each drill was used, and are so noted in the table.

### 4. Turning Data on Fiberglass

The turning tests were performed on a G-10 fiberglass cylinder, 8 inches in diameter. The lathe used was a Lodge-Shipley "Answer Lathe". Thirty-two inserts, 16 of which were coated with 0.3 mil of TiB<sub>2</sub>, were tested under various conditions summarized in Table A-6. The inserts used were grade CY14 tungsten carbide cobalt-based square tools from Wendt-Sonic/Unimet, style SNMP643E. Inserts 1, 3, 7, 17, 19, and 23 were used on the curved surface of the fiberglass cylinder by turning to a smaller diameter, while the other inserts were positioned for facing the fiberglass cylinder. A different spindle speed was chosen for each of the eight cutting points of the inserts with surface cutting speeds ranging from 52 sfm to 1498 sfm. As described before, the wear land on each cutting point was measured microscopically, and all tool-like data were based on a wear land of 0.02 inch. The amounts of material removed for each cutting point of the insert at various surface speeds were added together to obtain the last column in Table A-6, "Total Material Removed".

### 5. End Mill Tests on Fiberglass

Twenty-four high-speed steel end mills, 12 of them coated with 0.3 mil of TiB<sub>2</sub>, were tested. These end mills were 1/2-inch-diameter, single-end, straight shank. A groove in a G-10 fiberglass sheet, 1 inch thick was milled to a depth of 1/2 inch at a feed rate of 1.5 in./min. Table A-7 lists the material removed by each end mill for 0.02 inch wear.

## VI. ANALYSIS OF TOOL TEST DATA

In the type of test used to determine the value of a tool coating, a relatively large number of variables are involved that may affect performance. Since some of these variables may have a large effect and others a small effect, we have used correlation and the analysis of variance to determine the significant effects on the performance of a tool. Correlation is a measure of the tendency of two parameters to vary together in accord with a linear relationship. The mathematical models written for each set of tests presented here contain all the variables that can be controlled during the test. The technique of analysis of variance makes it possible to test the relevance of the factors in the model and to determine if they



interact, that is, if a combination of factors are relevant. It also provides an estimate of the variance in performance due to variables that are not controlled in the test. By comparing the total variance explained by the controlled variables with the residual variance, we know how much of the variation is due to the controlled variables and how much is due to unknown variables. If the residual is small, the model must contain all relevant variables and when the residual is large we know that some factors that affect the performance were left out of the model.

The following sections on each of the broad classifications of tests performed will contain, first, an equation which is a mathematical model for the test that includes all the controlled variables and interactions between them, and a term for the uncontrolled factors. Secondly, a table is constructed which lists the performance values for each combination of variables. The performance value is obtained by averaging the amount of material removed for each set of data points. A set of data points consists of six values of material removed at various surface speeds for drills and end mills, one value for each drill or end mill. A set of data points for inserts consists of eight values of material removed at various surface speeds, one value for each cutting edge on the insert. This is followed, for each classification of test, by another table which lists the statistical data for the analysis of variance. This table contains the sources of variation which are the variables that were controlled during the test, and the degrees of freedom for each variable which is the number of levels for that variable minus one. The table then shows the sum of squares which is a measure of the difference between the grand mean (in the complete set of experiments) and the mean values for these tests for which the factors A, B, etc., are present. The mean squares are the sum of squares divided by the degrees of freedom.

The test of significance, the F ratio, determines if the mean of the population of performance values that is of interest (e.g., all values in which the tool is coated) is significantly different from the mean of the entire population of performance values. This is calculated by forming the ratio of the mean squares for the factor considered and the residual, and is presented in the last column of the tables. If this value exceeds a critical value found in statistical tables (see, e.g., "Probability and Statistics for Engineers" by I. Miller and J. Freund, Prentice-Hall, 1965) then the analysis of variance indicates that the variable is significantly different.

## 1. Drilling Tests on Steel

The two-level variable factors were: A, workpiece hardness; B, TiB<sub>2</sub>-coated or uncoated; C, feed rate, 0.0015 in./rev or 0.003 in./rev; and D, dry or Lusol coolant. The model is

$$Y_{ijkl} = \mu + A_i + B_j + C_k + D_l + G_{ijkl}$$

where  $Y_{ijkl}$  is the performance value in cubic inches removed from the substrate for 0.005-inch wear on the drill. Table 16 lists the average value of this performance value for drills tested at six different surface speeds for each combination of variable factors.

Table 17 lists the sources of variation and the statistical information required to determine if the effects are significant. For the degrees of freedom

Table 16. DRILLING TESTS ON STEEL —  
MATERIAL REMOVED (CUBIC INCH)

	b <sub>0</sub>				b <sub>1</sub>			
	a <sub>0</sub>		a <sub>1</sub>		a <sub>0</sub>		a <sub>1</sub>	
	c <sub>0</sub>	c <sub>1</sub>	c <sub>0</sub>	c <sub>1</sub>	c <sub>0</sub>	c <sub>1</sub>	c <sub>0</sub>	c <sub>1</sub>
d <sub>0</sub>	0.739	0.585	0.392	0.347	0.495	0.066	0.033	0.025
d <sub>1</sub>	0.999	0.703	0.302	0.180	0.981	0.808	0.082	0.033

Legend

A. Workpiece Hardness	a <sub>0</sub> - Soft	a <sub>1</sub> - Hard
B. Coating	b <sub>0</sub> - Uncoated	b <sub>1</sub> - Coated
C. Feed Rate	c <sub>0</sub> - 0.0015 in./rev	c <sub>1</sub> - 0.003 in./rev
D. Coolant	d <sub>0</sub> - Dry	d <sub>1</sub> - Luso1 Coolant

Table 17. DRILLING TESTS ON STEEL —  
ANALYSIS OF VARIANCE

Source of Variation	D.F.	Sum of Squares	Mean Squares	F Ratio
Treatment				
A	1	1.00	1.00	50.00
B	1	0.19	0.19	19.00
C	1	0.10	0.10	13.09
D	1	0.12	0.12	15.89

(D.F.) in these variables the F ratio must be greater than 10:1 in order to be significant at the 95 percent probability level. The results indicate the most important factor to be workpiece hardness, with the harder workpiece causing a shorter tool life. The second most important effect is the coating since coated tools on the average did not perform as well as the uncoated tools. Both the feed rate and the cooling had a significant effect on tool life also.

Metallurgical examination of polished cross sections of coated and uncoated drills show some carbide particle growth caused by tempering at deposition temperature. Figure 15 is a 500X photomicrograph showing coated and uncoated drill substrates. The metallurgical damage shown in Figure 15 is enough to account for the loss in substrate strength and subsequent failure of coated drills.

## 2. Turning Data on Steel — Statistical Analysis

In these tests the factors were workpiece hardness, depth of cut, feed rate, cooling, and finally whether the tool was coated with TiB<sub>2</sub> or not. These factors are all two-level factors and the model is

$$Y_{ijklm} = \mu + A_i + B_j + C_k + D_l + E_m + G_{ijklm} + \text{interactions}$$

where  $Y_{ijklm}$  is the measured performance of the tool in cubic inches removed for 0.02-inch wear produced. The two-level factors A through E represent workpiece hardness, depth of cut, feed rate, coolant, and coating. The model contains interactions of possible significance that will be discussed later.

The performance data for all the inserts from Table A-2, is reproduced in Table 18 except that the average values for material removed by each of the eight cutting edges of the insert are used instead of the total value. A preliminary review of Table 18 would indicate that in general the performance values under e<sub>1</sub>, for the TiB<sub>2</sub>-coated tools, are higher than those under e<sub>0</sub>, for tools without the coating. Table 19 is constructed in order to show that this difference is statistically significant. The sum of squares is shown for each of the treatments, along with the mean squares which are the same since the number of degrees of freedom is 1 in all cases. If an F ratio greater than 4.17 is calculated, then the probability of significance would be at the 95 percent level. The calculated F ratios which show significant effects are the depth of the cut, the TiB<sub>2</sub> coating and the soluble oil coolant. Among the interactions the combination of feed rate and cooling produced significantly better tool life.





a. Uncoated (Transverse)



b. Uncoated (Longitudinal)



c. Coated (Longitudinal)



d. Coated (Transverse)

Figure 15. Photomicrographs of uncoated and coated drill steel. Mag. 500X

Table 18. TURNING DATA ON STEEL —  
MATERIAL REMOVED (CUBIC INCH)

	e <sub>0</sub>				e <sub>1</sub>			
	d <sub>0</sub>		d <sub>1</sub>		d <sub>0</sub>		d <sub>1</sub>	
	c <sub>0</sub>	c <sub>1</sub>	c <sub>0</sub>	c <sub>1</sub>	c <sub>0</sub>	c <sub>1</sub>	c <sub>0</sub>	c <sub>1</sub>
a <sub>0</sub>	0.71	0.91	1.59	0.91	0.79	1.04	1.89	2.68
b <sub>0</sub>	a <sub>1</sub>	0.52	1.53	2.06	1.26	0.67	1.18	1.87
a <sub>1</sub>	0.77	0.92	1.73	1.55	1.05	1.13	6.45	2.50
b <sub>1</sub>	a <sub>1</sub>	1.06	2.11	1.79	1.64	1.08	1.99	2.01
a <sub>1</sub>							2.45	

Legend

A. Hardness	a <sub>0</sub> - Soft	a <sub>1</sub> - Hard
B. Depth of Cut	b <sub>0</sub> - 0.01 in.	b <sub>1</sub> - 0.02 in.
C. Feed Rate	c <sub>0</sub> - 0.01 in./rev	c <sub>1</sub> - 0.02 in./rev
D. Cooling	d <sub>0</sub> - Uncooled	d <sub>1</sub> - Cooled
E. Coating	e <sub>0</sub> - Uncoated	e <sub>1</sub> - Coated

Table 19. TURNING DATA ON STEEL —  
ANALYSIS OF VARIANCE

Source of Variation	D.F.	Sum of Squares	Mean Squares	F Ratio
<u>Treatment</u>				
A	1	0.10	0.10	0.17
B	1	2.55	2.55	4.26
C	1	0.01	0.01	0.02
D	1	8.51	8.51	14.21
E	1	2.70	2.70	4.51
<u>Interaction</u>				
AB	1	0.14	0.14	0.23
AC	1	1.13	1.13	1.89
AD	1	1.74	1.74	2.90
AE	1	1.80	1.80	3.00
BC	1	0.22	0.22	0.36
BD	1	0.39	0.39	0.65
BE	1	0.74	0.74	1.24
CD	1	2.52	2.52	4.21
CE	1	0.11	0.11	0.18
DE	1	2.26	2.26	3.77
<u>Residual</u>	16	9.58	0.60	
<u>Total</u>	31	34.51		

Inserts 33 and 42 from Table A-3 were not included in this analysis, since the inserts used were of a different type, a nickel-bonded rather than cobalt-bonded carbide. These tests were not extensive enough to warrant an analysis of variance; however, the results were many times better for TiB<sub>2</sub>-coated inserts than for the uncoated ones. The best performance for the TiB<sub>2</sub>-coated inserts was at 600 sfm on a hard workpiece where 0.114 cu in. was removed compared to 0.014 cu in. for the uncoated tool under the same conditions. The statistical analysis of inserts 43 to 56 in which performance data were summarized in Table A-4 was not attempted since the tests were not a complete set. However, an examination of the data indicates that changing the lead angle from 0° to 20° was a negative factor on performance of both coated and uncoated tools, and did not change their relative performance significantly.

### 3. Drilling Tests on Fiberglass

The sources of variation in these tests were A, coating; B, feed rate; and C, coolant. The model is

$$Y_{ijk} = \mu + A_i + B_j + C_k + (AB)_{ij} + (AC)_{ik} + (BC)_{jk} + G_{ijk}$$

where  $Y_{ijk}$  is the performance value, the average amount of material removed by a drill at six different surface speeds for one combination of the factors. The interaction effects  $(AB)_{ij}$  and  $(AC)_{ik}$  were included in the model since two of them were found to be significant, and will be discussed later.

The performance values are shown in Table 20, and even a casual comparison of the lower half of the table, the coated drill data, with the uncoated data in the upper half, would indicate that the coating significantly improves the performance of these drills. Table 21 shows this to be true, with the F ratio for the coating source of variation a very large 1006.11. The feed rate and the coolant used also produced significant effects on performance. Both interactions that contain the coating effect are significant.

Table 20. DRILLING TESTS ON FIBERGLASS —  
MATERIAL REMOVED (CUBIC INCH)

		b <sub>0</sub>	b <sub>1</sub>	b <sub>2</sub>
a <sub>0</sub>	c <sub>0</sub>	0.43	0.42	0.75
	c <sub>1</sub>	0.34	0.46	0.63
	c <sub>2</sub>	0.23	0.36	0.45
a <sub>1</sub>	c <sub>0</sub>	2.89	5.24	4.61
	c <sub>1</sub>	4.05	5.47	5.82
	c <sub>2</sub>	1.39	3.16	3.21

Legend

A. Coating	a <sub>0</sub> - Uncoated	a <sub>1</sub> - Coated
B. Feed Rate	b <sub>0</sub> - 0.0015 in./rev	b <sub>1</sub> - 0.003 in./rev
	b <sub>2</sub> - 0.006 in./rev	
C. Coolant	c <sub>0</sub> - Uncooled	
	c <sub>1</sub> - I, Luso1	
	c <sub>2</sub> - II, Soluble Oil	

Table 21. DRILLING TESTS ON FIBERGLASS —  
ANALYSIS OF VARIANCE

Source of Variation	D.F.	Sum of Squares	Mean Squares	F Ratio
<u>Treatment</u>				
A	1	56.07	56.07	1006.11
B	2	3.96	1.98	35.51
C	2	5.56	2.78	49.90
<u>Interaction</u>				
AB	2	2.71	1.36	24.34
AC	2	4.38	2.19	39.33
BC	4	0.11	0.03	0.49
<u>Residual</u>	4	0.22	0.06	
<u>Total</u>	17	73.02		

#### 4. Turning Tests on Fiberglass

There are five sources of variation for these tests, rake angle, depth of cut, feed rate, coolant, and coating. Table 22 contains the performance data in the form of the total amount of material removed from the workpiece by each insert. The analysis of variance, Table 23, indicates that only two F ratios show significant effects. The larger depth of cut had a beneficial effect on performance and the interaction of the soluble oil coolant and the coating was also significant.

The model is

$$Y_{ijklm} = \mu + A_i + B_j + C_k + D_l + E_m + (DE)_{lm} + G_{ijklm}$$

where  $(DE)_{lm}$  is the coolant-coating interaction.

Table 22. TURNING DATA ON FIBERGLASS —  
MATERIAL REMOVED (CUBIC INCH)

		e <sub>0</sub>				e <sub>1</sub>			
		d <sub>0</sub>		d <sub>1</sub>		d <sub>0</sub>		d <sub>1</sub>	
		c <sub>0</sub>	c <sub>1</sub>	c <sub>0</sub>	c <sub>1</sub>	c <sub>0</sub>	c <sub>1</sub>	c <sub>0</sub>	c <sub>1</sub>
b <sub>0</sub>	a <sub>0</sub>	123.8	48.7	40.3	112.1	20.1	41.0	22.5	39.3
	a <sub>1</sub>	47.3	13.5	18.6	30.8	21.8	13.1	24.5	171.8
b <sub>1</sub>	a <sub>0</sub>	118.6	95.6	103.7	43.4	122.5	80.7	29.8	27.5
	a <sub>1</sub>	175.8	294.8	68.0	67.8	32.2	11.8	114.0	170.5

Legend

A. Rake Angle	a <sub>0</sub> - 5°	a <sub>1</sub> - 10°
B. Depth of Cut, in.	b <sub>0</sub> - 0.050	b <sub>1</sub> - 0.075
C. Feed Rate, in./min	c <sub>0</sub> - 0.010	c <sub>1</sub> - 0.020
D. Cooling	d <sub>0</sub> - Uncooled	d <sub>1</sub> - Cooled
E. Coating	e <sub>0</sub> - Uncoated	e <sub>1</sub> - Coated

Table 23. TURNING DATA ON FIBERGLASS —  
ANALYSIS OF VARIANCE

Source of Variation	D.F.	Sum of Squares	Mean Squares	F Ratio
<u>Treatment</u>				
A	1	21.70	21.70	0.39
B	1	251.16	251.16	4.54
C	1	18.29	18.29	0.33
D	1	12.86	12.86	0.23
E	1	121.56	121.56	2.20
<u>Interaction</u>				
AB	1	107.49	107.49	1.94
AC	1	59.70	59.70	1.07
AD	1	36.36	36.36	0.65
AE	1	16.63	16.63	0.30
BC	1	3.50	3.50	0.06
BD	1	77.59	77.59	1.40
BE	1	47.60	47.60	0.86
CD	1	44.34	44.34	0.80
CE	1	21.17	21.17	0.38
DE	1	267.67	267.67	4.83
<u>Residual</u>	16	886.03	55.37	
<u>Total</u>	31	1993.69		



## 5. End Mill Tests on Fiberglass

Only two sources of variation were permitted in these tests: coating and cooling. The model is

$$Y_{ij} = \mu + A_i + B_j = (AB)_{ij} + G_{ij}.$$

The performance data listed in Table 24 are the average amounts of material removed by six end mills for each of the four conditions. Table 25 shows that the A treatment, that is, the coating, has a significant positive effect on the wear life of the tool.

Table 24. END MILL TESTS — MATERIAL REMOVED (CUBIC INCH)

	$b_0$	$b_1$
$a_0$	1.98	3.14
$a_1$	4.40	6.64

Legend

A. Coating  $a_0$  - Uncoated  $a_1$  - Coated  
B. Coolant  $b_0$  - Uncooled  $b_1$  - Cooled

Table 25. END MILL TESTS — ANALYSIS OF VARIANCE

Source of Variation	D.F.	Sum of Squares	Mean Squares	F Ratio
<u>Treatment</u>				
A	1	52.87	52.87	8.13
B	1	17.02	17.02	2.62
<u>Interaction</u>	1	1.66	1.66	0.25
<u>Residual</u>	20	130.04	6.50	
<u>Total</u>	23	201.60		

## 6. Statistical Data Summary and Discussion

The coating of high-speed steel drills with  $TiB_2$  was not effective when the workpiece was steel, but was highly effective when the workpiece was fiberglass. Table 26 shows the estimates of effects for drilling tests for each variable. These are measures of how the mean values were changed by the various effects.

Table 26. ESTIMATE OF EFFECTS FOR DRILLING TESTS

Test	Mean	<u>Coating</u>		<u>Hardness</u>		<u>Feed Rate (in./rev)</u>			<u>Coolant</u>		
		Coated	Uncoated	Soft	Hard	0.0015	0.003	0.006	Dry	Lusol	Soluble Oil
Drilling Steel	0.423	-0.11	+0.11	0.25	-0.25	0.08	-0.08	-	-0.09	0.09	-
Drilling Fiberglass	2.22	1.77	-1.77	-	-	-0.66	0.30	0.36	0.17	0.58	-0.75

The mean performance value for all drilling tests on steel was 0.423 cu in. removed and for all drilling tests on fiberglass was 2.22 cu in. removed. The coated drills did only 59 percent as well as uncoated drills when used on steel, but did 887 percent better when used on fiberglass. For both workpieces, cooling with Lusol improved tool performance, and the higher feed rates were better on fiberglass while the lower feed rate was better on steel. As one would expect, the harder steel workpiece was more difficult to drill.

These results were understood clearly when it was determined that the coating process softened the high-speed steel drills while the  $TiB_2$  was being deposited. Drills whose coatings were removed did not perform as well as uncoated drills



(drills 137 and 138 in Table A-1) apparently because of a loss of strength by tempering at the 650 C process temperature. In the case of the fiberglass workpiece, the high strength of the drill substrate is not needed since the material is not as strong. However, the fiberglass is extremely abrasive and appears to wear tools by grinding them down with its hard abrasive grit produced during the drilling operation. The effectiveness of the coating in withstanding this abrasive wear is due to its extreme hardness. Table 27 lists a few of the interaction effects which will determine the best conditions of use for the coated drills in fiberglass drilling operations.

Table 27. INTERACTION EFFECTS FOR DRILL TESTS ON FIBERGLASS

Condition	Mean	Effect	% of Mean
Coated, 0.0015 in./rev Feed Rate	3.99	-0.092	95
Coated, 0.003 in./rev Feed Rate	3.99	-0.553	76
Coated, 0.006 in./rev Feed Rate	3.99	0.645	139
Coated, Uncooled	3.99	0.092	105
Coated, Lusol Coolant	3.99	0.553	132
Coated, Soluble Oil	3.99	-0.645	72

The coated drills that were used with the highest feed rate, 0.006 in./sec and with Lusol coolant, produced the longest tool life.

The carbide inserts used in the turning tests represent an altogether different substrate material for the  $TiB_2$  coating than high-speed steel. The carbide did not seem to degrade in performance because of the process since it is not temperature sensitive; and since it is so much harder than high-speed steel, the difference in abrasive wear between coated and uncoated carbide is much smaller. Table 28 lists the turning tests on steel and fiberglass and the effect of the variables on the mean tool life.

Table 28. ESTIMATE OF EFFECTS FOR TURNING TESTS

Test	Mean	Coating		Coolant		Feed Rate (in./rev)		Depth of Cut (in.)		Hardness	
		Coated	Uncoated	Dry	Lusol	Low	High	Small	Large	Soft	Hard
Turning Steel	1.61	0.29	-0.29	-0.52	0.52	0.02	-0.02	-0.28	0.28	0.06	-0.06
Turning Fiberglass	9.15	-1.95	1.95	0.63	-0.63	-0.76	0.76	-2.80	2.80	-	-

The coated inserts performed 44 percent better than uncoated inserts when turning steel. The best conditions were Lusol coolant, low feed rate, and large depth of cut. The harder steel workpiece shortened tool life relative to the softer workpiece, as expected. The turning of fiberglass with carbide inserts is difficult except at very low surface speeds, and the coating did not improve the performance of the carbide.

The performance of the steel end mills was improved by the application of  $TiB_2$  coating as shown in Table 29.

The coated end mill performed 215 percent better than the uncoated tool and the coolant also improved performances. This test is another illustration of the effect of applying the coating to steel tools used on fiberglass, that is, a much improved tool life.

Table 29. ESTIMATE OF EFFECTS FOR END MILL TESTS

Mean	Coating		Coolant	
	Coated	Uncoated	Dry	Lusol
4.05	1.48	-1.48	-0.84	0.84

## VII. COST COMPARISON

The cost of machining a workpiece is the product of the interaction of many factors, some of which can only be estimated. The first estimate is to determine if minimum cost per piece or maximum productivity is the priority criterion. This difference between minimum cost tool life and maximum production tool life is determined by the cutting speed that can be attained with the tool. In our estimates we have used the high surface speeds to compare costs for the maximum production tool life.

### 1. Drilling Tests

Although the coated drills did only 75 percent as well as uncoated drills on 4340 steel, the coating improved tool life an average of 887 percent for drills used on fiberglass or about a factor of 9. This improvement in tool life was for surface speeds ranging from 15 sfm to 185 sfm. For this comparison wage rates and tool costs were taken from Weller<sup>16</sup> who estimates an average hourly wage rate of \$4.00, drill cost of about \$0.40 (10 percent of the average hourly wage rate) and overhead of 650 percent of hourly wage rates, or about \$26.00 per hour.

The first step in our economic analysis of TiB<sub>2</sub>-coated drills was to estimate the cost of plating a large number of drills. We make the assumption that a plating facility accommodates about 50 lb of electrolyte with the capability of plating 200 1/4-in. drills at one time. This facility is used for 12 weeks for 5 runs per day for a total production run of 60,000 drills. Table 30 lists the costs associated with the setting up of this facility, its maintenance, and production runs. Also included are the cost of the materials used and the amortization of equipment. The total cost to plate 60,000 drills, using a labor plus overhead charge of \$30 per hour, is \$18,834 or \$0.31 per drill.

Table 30. COST ANALYSIS OF COATED DRILLS

		Hour	Cost
1. Labor			
A. Bath Setup		40	\$ 1,200
B. Maintenance		12	360
C. Production		480	14,400
2. Materials			
A. Bath, Crucible, Etc.			1,000
B. Titanium, Boron on Drills			40
3. Energy Cost			
A. Furnace Heating		4 kW	403
\$0.05/kW hr (8,064 kW hr)			
B. Plating Energy Cost		2,000 A-hr	1
4. Equipment Amortization	Cost	Lifetime (yr)	\$/12 wk
A. Plating Facility	\$5,000	1	1,154
B. Furnace	3,000	5	138
C. Power Supply, Etc.	3,000	5	138
5. Total Cost:	\$18,834 (\$0.31/Drill)		

16. WELLER, E. J. *Tool Economics*. Society Manufacturing Engineers, Paper MR71-939, 1973.

In order to compare the cost of drilling with coated and uncoated drills, estimates of the cost of a drilling operation were made using two prices for the drills, \$0.40 for uncoated drills and \$0.71 for coated drills. The drilling operation consists of drilling 1/4-in. holes through each of one thousand 1/2-in. workpieces made of G-10 fiberglass 2 in. wide and 3 in. long. It was assumed that the work was done on an upright drilling machine equipped with power feed and various drilling speeds and that the piece was held at the drilling station in an air-operated vise. The operation starts with the operator picking the piece from a bin, locating the piece, drilling the hole, and removing the piece. We have estimated (see, for example, "Estimating Machining Costs", C. W. S. Parsons, McGraw-Hill, 1957) these operations to take a total of 0.3 minute and changing the drill to take 0.1 minute. Table 31 summarizes the model drilling operation. The feed rate is 0.006 in./rev, which, at the given rotational speed of the machine (2840 rpm), results in a surface speed of 185 sfm. As shown in Table A-5, at this speed the uncoated drill (No. 61) was able to drill 13 holes, and thus the operator would change drills after 13 pieces and use a total of 77 drills to do all 1000 parts. The coated drill (No. 66) was able to drill 120 holes and therefore only 9 drills would be needed. Table 32 lists the time and dollars required for tools and labor with each type of drill. Tool costs for the coated drills are just 21 percent of the cost of uncoated drills; and time is reduced, therefore labor costs are reduced also. The short setup time, however, reduces the overall cost advantage of using coated drills to approximately 3 percent.

In a machining situation where setup time is longer, the savings are even greater in labor cost as illustrated in Table 33, in which the setup time has been increased to 0.3 minute. In this case an overall savings of \$642.17 or approximately 7 percent results from the use of the coated drills instead of uncoated drills. Thus it can be seen that the use of coated drills reduces tooling

Table 31. MODEL DRILLING OPERATION

Operation Elements	Time (min)	Feed (in./rev)	Travel (in.)	RPM	SFM	Coolant	Number of Parts
Positioning, Drilling, and Depositioning	0.3	0.006	0.5	2,840	185	Lusol	1,000
Changing Tool	0.1						

Table 32. DRILLING COST COMPARISON, SHORT SETUP TIME

	Time (min)			Cost		
	Operation	Tool Changing	Total	Labor \$30/hr	Tool	Total
Uncoated Drill	300	7.7	307.7	\$9,231	\$30.80	\$9,261.80
Coated Drill	300	0.9	0.9	9,027	6.39	9,033.39

Table 33. DRILLING COST COMPARISON, LONG SETUP TIME

	Time (min)			Cost		
	Operation	Tool Changing	Total	Labor \$30/hr	Tool	Total
Uncoated Drill	300	23.1	323.18	\$9,693	\$30.80	\$9,723.80
Coated Drill	300	2.7	302.7	9,081	6.39	9,087.39

cost for drilling fiberglass, may also provide a significant reduction in labor costs, especially if setup time and tool changing time are large compared to the operation time.

## 2. Turning Tests

Although the tool life in turning fiberglass was not improved by the coating, the tests on the turning of steel showed a significant increase in tool life for coated inserts. The data for all cutting edges for inserts 13 and 14 are compared in Table 34. The total material removed for the eight edges of each insert is listed in Table A-2. This test was conducted with soluble oil coolant at a depth of cut of 0.02 in., a feed rate of 0.0019 in./rev on the 4340 steel workpiece of hardness HRC 25. The cost was estimated for a turning operation using 5/8-in.-square disposable carbide inserts that are coated and uncoated for comparison. The assumed operation was a straight 0.02-in.-deep cut on a 3.5-in.-diameter steel cylinder, each cut 1 in. long. Costs are based on processing 1000 pieces. Table 35 summarizes the conditions and time estimates for each operation.

Each square insert has eight cutting edges and costs \$1.60. Therefore, the cost per cutting edge is \$0.20. The coated inserts were assumed to cost the same to plate as the drills discussed in the preceding section, i.e., \$0.31 each, thus the cost per cutting edge of the coated insert is \$0.24. The performance values used in Table 36 were obtained from Table 34 by averaging the three lowest spindle speeds performance values. These three were chosen because they were the most effective from a tool utilization standpoint. Since 0.2 cu in. is removed from each

Table 34. TOOL LIFE COMPARISON — MATERIAL REMOVED (CUBIC INCH)  
BY EACH CUTTING EDGE

Insert	Coating	RPM/123	169	241	342	472	672	912	1263
13	Uncoated	0.422	0.174	0.294	0.296	0.223	0.113	0.116	0.092
14	Coated	1.57	3.62	0.202	0.306	0.236	0.169	0.156	0.187

Table 35. MODEL TURNING OPERATION

Operation Elements	Time (min)	Feed Rate (in./rev)	Travel (in.)	Depth of Cut (in.)	RPM	SFM	Coolant	Number of Parts
Positioning Cylinder, Turning, and Removing Cylinder	3.2	0.0019	1	0.02	170	155	Soluble Oil	1,000
Changing Tool	0.2							

Table 36. TURNING COST COMPARISON

	Time (min)			Cost		
	Operation	Tool Changing	Total	Labor \$30/hr	Tool	Total
Uncoated Insert	3,200	133.4	3,333.4	\$100,002	\$133.40	\$100,135.40
Coated Insert	3,200	22.2	3,222.2	96,666	26.64	96,692.64



part, 83.3 uncoated inserts will be needed, with 667 cutting edge changes, compared to 13.9 coated inserts required, with 111 cutting edge changes.

Thus, the use of coated inserts results in a savings of \$3,442.76, or approximately 3 percent. The total cost for coated inserts is only 20 percent of the total cost for uncoated inserts because of the longer life of the coated tools.

### 3. End Mill Tests on Fiberglass

As indicated in Table 7, a coated end mill 14 removed 2.00 cu in. of workpiece for the same wear as uncoated end mill 13 that removed 0.54 cu in. Parameters for a sample milling operation selected for cost comparison are listed in Table 37. In this operation a groove is cut 1/2 in. wide, 1/2 in. deep, and 1 in. long in a fiberglass work piece 2 in. wide, 1 in. long, and 1 in. thick.

The end mill must remove 0.25 cu in. from each part; therefore, 463 uncoated end mills will be needed compared to 125 coated end mills. We will assume uncoated end mills can be obtained for \$1.00 each and that plating will add \$0.31 each to their cost. Table 38 shows the costs associated with our model milling operation.

The coated end mills allowed a saving of \$2,327.25 on the total cost (approximately 2.5 percent), and total costs of coated end mills were only 35 percent of the total cost of uncoated end mills.

Table 37. MODEL MILLING OPERATION

Operation Elements	Time (min)	Feed Rate (in./rev)	Travel (in.)	Coolant	Depth of Cut (in.)	RPM	SFM	Number of Parts
Positioning, Milling, and Removing Part	1.5	1.5	1	Lusol	0.5	1,163	153	1,000
Changing Tool	0.2							

Table 38. MILLING COST COMPARISON

	Time (min)			Cost		
	Operation	Tool Changing	Total	Labor \$30/hr	Tool	Total
Uncoated End Mill	1,500	92.6	1,592.6	\$47,778	\$463.00	\$48,241.00
Coated End Mill	1,500	25.0	1,525.0	45,750	163.75	45,913.75

## VIII. CONCLUSIONS

1. A  $\text{TiB}_2$  coating plated on steel offers a tough, very hard, adherent layer, resistant to spalling and surface wear.

2. The  $\text{TiB}_2$  coating improves the performance of 1/4" high-speed steel drills by an average 887 percent when used on fiberglass.

3. The  $\text{TiB}_2$  coating improves the performance of 1/2" high-speed steel end mills by an average 215 percent when used on fiberglass.

4. The  $\text{TiB}_2$  coating improves the performance of disposable 5/8" carbide inserts by an average 44 percent when used on 4340 steel.

5. These improvements can result in significant cost savings in typical production machining operations.

6. The  $\text{TiB}_2$  coating did not improve the performance of carbide inserts used on fiberglass, or high-speed steel drills used on steel.

7. The loss in performance of the  $\text{TiB}_2$ -coated high-speed steel drills used on steel was at least partially due to metallurgical changes in the substrate caused by the high process temperature and resulting in a loss of strength.

8. The  $\text{TiB}_2$  coating appears to offer a degree of protection against laser irradiation damage.

#### IX. ACKNOWLEDGMENTS

We wish to thank Mr. J. J. Comer of the Air Force Cambridge Research Laboratories for the electron diffraction study and Dr. Luigina C. Sogliers of UTRC for the mathematical analysis. Many of our other colleagues at AMMRC and UTRC offered valuable advice and suggestions, particularly Dr. Homer F. Priest and Mr. Robert Fitzpatrick.

# APPENDIX A. TOOL TEST DATA FOR STEEL AND FIBERGLASS WORKPIECES

Table A-1. DRILL TEST DATA ON STEEL

Drill #	A	B	C	D	sfm	(cu in.)	# Holes
						Material Removed	
30	0	0	1	0	246	.13	5
43	0	0	1	0	185	.15	6
31	0	0	1	0	153	.88	36
44	0	0	1	0	124	1.08	44
32	0	0	1	0	94	1.45	60
45	0	0	1	0	77	2.31	94
29	0	1	1	0	246	.098	4
46	0	1	1	0	185	.098	4
28	0	1	1	0	153	.64	26
47	0	1	1	0	124	.20	8
27	0	1	1	0	94	1.52	62
48	0	1	1	0	77	1.67	68
19	0	0	0	0	185	.074	3
21	0	0	0	0	153	.59	24
18	0	0	0	0	124	.64	26
17	0	0	0	0	77	.69	28
16	0	0	0	0	46	.54	22
20	0	0	0	0	22	1.91	78
7	0	1	0	0	246	.074	3
10	0	1	0	0	185	.049	2
11	0	1	0	0	124	.22	9
5	0	1	0	0	94	.61	25
8	0	1	0	0	46	.98	40
9	0	1	0	0	29	1.57	64
37	0	0	1	1	246	.025	1
53	0	0	1	1	185	.25	10
36	0	0	1	1	153	.91	37
52	0	0	1	1	124	.32	13
38	0	0	1	1	94	1.52	62
51	0	0	1	1	77	2.87	117

Table A-1 (Continued)

Drill #	A	B	C	D	sfm	(cu in.) Material Removed	# Holes
41	0	1	1	1	246	.42	17
55	0	1	1	1	185	.15	6
42	0	1	1	1	153	.98	40
54	0	1	1	1	124	.32	13
39	0	1	1	1	94	2.57	105
56	0	1	1	1	77	.42	17
64	0	0	2	0	246	.098	4
62	0	0	2	0	185	.05	2
66	0	0	2	0	153	1.20	49
60	0	0	2	0	124	.52	21
69	0	0	2	0	94	1.40	57
58	0	0	2	0	77	1.64	67
65	0	0	2	1	246	.074	3
63	0	0	2	1	185	.025	1
67	0	0	2	1	153	.76	31
61	0	0	2	1	124	.25	10
68	0	0	2	1	94	.61	25
59	0	0	2	1	77	2.33	95
23	0	0	0	1	77	.025	1
22	0	0	0	1	46	1.23	50
15	0	0	0	1	46	.17	7
26	0	0	0	1	22	.93	38
24	0	0	0	1	22	.12	5
12	0	1	0	1	124	.025	1
13	0	1	0	1	77	.025	1
14	0	1	0	1	46	.15	6
86	1	0	0	0	22	.71	29
76	1	0	0	0	29	.78	32
85	1	0	0	0	46	.025	1
71	1	0	0	0	65	.74	30



Table A-1 (Continued)

Drill #	A	B	C	D	sfm	(cu in.) Material Removed	# Holes
84	1	0	0	0	77	.025	1
82	1	0	0	0	94	.074	3
88	1	1	0	0	15	.96	39
87	1	1	0	0	22	.25	10
74	1	1	0	0	29	.71	29
73	1	1	0	0	65	.049	2
83	1	1	0	0	77	.025	1
80	1	1	0	0	94	.098	4
78	1	0	0	1	10	.049	2
77	1	0	0	1	29	.025	1
72	1	0	0	1	65	.025	1
79	1	1	0	1	10	.025	1
75	1	1	0	1	29	.025	1
81	1	1	0	1	94	.025	1
94	1	0	1	0	22	.54	22
91	1	0	1	0	46	.34	14
106	1	0	1	0	65	.29	12
90	1	0	1	0	77	.20	8
104	1	0	1	0	94	.42	17
89	1	0	1	0	124	.025	1
98	1	1	1	0	15	.32	13
95	1	1	1	0	22	.34	14
108	1	1	1	0	29	.20	8
99	1	1	1	0	46	.12	5
107	1	1	1	0	65	.074	3
101	1	1	1	0	77	.025	1
93	1	0	1	1	22	.12	5
92	1	0	1	1	46	.025	1
105	1	0	1	1	65	.098	4

Table A-1 (Continued)

Drill #	A	B	C	D	sfm	(cu in.) Material Removed	# Holes
97	1	1	1	1	15	.025	1
96	1	1	1	1	22	.025	1
100	1	1	1	1	46	.049	2
111 Carbide	1	0	1	0	29	.29	12
103	1	0	1	0	46	.025	1
109	1	0	1	0	65	.15	6
102	1	0	1	0	77	.20	8
112	1	0	1	0	94	.25	10
118	1	0	1	0	153	.71	29
116 Carbide	1	1	1	0	29	.17	7
115	1	1	1	0	65	.12	5
114	1	1	1	0	94	.15	6
117	1	1	1	0	153	.22	9
120 Carbide	1	0	1	1	29	.025	1
110	1	0	1	1	65	.074	3
113	1	0	1	1	94	.025	1
119	1	0	1	1	153	.049	2
121 Carbide	1	1	1	1	29	.025	1
122	1	1	1	1	65	.025	1
123	1	1	1	1	94	.025	1
136	1	0	2	0	15	.54	22
133	1	0	2	0	22	.34	14
130	1	0	2	0	29	.27	11
127	1	0	2	0	65	.25	10
132	1	0	2	0	77	.049	2
124	1	0	2	0	94	.25	10
128	1	0	2	0	153	.074	3
135	1	0	2	1	15	.025	1
134	1	0	2	1	22	.049	2
131	1	0	2	1	29	.025	1

Table A-1 (Continued)

Drill #		A	B	C	D	sfm	(cu in.) Material Removed	# Holes
126		1	0	2	1	65	.025	1
125		1	0	2	1	94	.12	5
129		1	0	2	1	153	.025	1
137	Uncoated but	1	0	2	2	15	.049	2
138	Processed	1	0	2	2	22	.049	2

Legend:

	0	1	2
A - Hardness	Soft	Hard	
B - Feed Rate	0.003"/rev	0.006"/rev	
C - Coolant	Dry	Lusol	Soluble Oil
D - Coating	Uncoated	Coated	Uncoated but Processed

Table A-2. TURNING DATA ON STEEL

Exp. Cond.	a	b	c	d	e	(cu in.) Total Material Removed	Insert #
1	0	0	0	0	0	0.71	3
a	1	0	0	0	0	0.52	23
b	0	1	0	0	0	0.77	5
ab	1	1	0	0	0	1.06	21
c	0	0	1	0	0	0.91	1
ac	1	0	1	0	0	1.53	19
bc	0	1	1	0	0	0.92	7
abc	1	1	1	0	0	2.11	17
d	0	0	0	1	0	1.59	15
ad	1	0	0	1	0	2.06	31
bd	0	1	0	1	0	1.73	13
abd	1	1	0	1	0	1.79	29
cd	0	0	1	1	0	0.91	11
acd	1	0	1	1	0	1.26	27
bcd	0	1	1	1	0	1.55	9
abcd	1	1	1	1	0	1.64	25
e	0	0	0	0	1	0.79	4
ae	1	0	0	0	1	0.67	24
be	0	1	0	0	1	1.05	6
abe	1	1	0	0	1	1.08	22
ce	0	0	1	0	1	1.04	2
ace	1	0	1	0	1	1.18	20
bce	0	1	1	0	1	1.13	8
abce	1	1	1	0	1	1.99	18
de	0	0	0	1	1	1.89	16
ade	1	0	0	1	1	1.87	32
bde	0	1	0	1	1	6.45	14
abde	1	1	0	1	1	2.01	30
cde	0	0	1	1	1	2.68	12
acde	1	0	1	1	1	1.58	28
bcde	0	1	1	1	1	2.50	10
abcde	1	1	1	1	1	2.45	26

Legend:

	0	1
a - Hardness	Soft	Hard
b - Depth of Cut, in.	0.01	0.02
c - Feed Rate, in./rev	0.0019	0.0039
d - Coolant	Dry	Soluble Oil
e - Coating	Uncoated	Coated

Table A-3. TURNING DATA ON STEEL (NICKEL-BONDED INSERTS)

Experimental Condition						(cu in.) Total Material	Insert #
	a	b	c	d	e	Removed	
ad	1	0	0	1	0	0.21	35
abd	1	1	0	1	0	0.32	37
acd	1	0	1	1	0	0.19	33
abcd	1	1	1	1	0	0.37	39
ade	1	0	0	1	1	1.13	36
abde	1	1	0	1	1	0.72	38
acde	1	0	1	1	1	0.99	34
abcde	1	1	1	1	1	1.61	40
abc	1	1	1	0	0	0.23	41
abce	1	1	1	0	1	0.67	42

Table A-4. TURNING DATA ON STEEL (20° LEAD ANGLE)

Experimental Condition						(cu in.) Total Material	Insert #
	a	b	c	d	e	Removed	
a	1	0	0	0	0	0.34	47
b	0	1	0	0	0	0.14	55
c	0	0	1	0	0	0.92	51
ac	1	0	1	0	0	1.40	43
bc	0	1	1	0	0	0.67	53
ae	1	0	0	0	1	0.45	48
ce	0	0	1	0	1	1.11	52
be	0	1	0	0	1	0.50	56
abc	1	1	1	0	0	.62	49
bce	0	1	1	0	1	.51	54
ace	1	0	1	0	1	.59	44
abce	1	1	1	0	1	.62	50
ace*	1	-	1	0	1	.10	46
ac*	1	-	1	0	0	.10	45

Legend:

	0	1
a - Hardness	Soft	Hard
b - Depth of Cut, in.	0.01	0.02
c - Feed Rate, in./rev	0.0019	0.0039
d - Coolant	Dry	Soluble Oil
e - Coating	Uncoated	Coated

\*Depth of Cut = 0.03"

Table A-5. DRILL TESTS ON FIBERGLASS

Drill #	a	b	c	sfm	# Holes	(cu in.) Material Removed
1	0	0	0	46	23	0.56
2	0	1	0	46	19	.47
3	0	2	0	46	39	.96
4	0	2	0	77	33	.81
5	0	1	0	77	20	.49
6	0	0	0	77	24	.59
7	0	0	0	124	13	.32
8	0	1	0	124	16	.39
9	0	2	0	124	24	.59
10	0	2	0	185	18	.44
11	0	1	0	185	12	.29
12	0	0	0	185	8	.20
13	0	0	0	22	22	.54
14	0	1	0	22	32	.78
15	0	2	0	22	52	1.27



Table A-5 (Continued)

Drill #	a	b	c	sfm	# Holes	(cu in.) Material Removed
16	0	2	0	15	65	1.59
17	0	1	0	15	44	1.08
18	0	0	0	15	17	0.42
19	1	0	0	15	32	0.78
20	1	0	0	22	90	2.21
21	1	1	0	22	175	4.29
22	1	2	0	22	120	2.94
23	1	2	0	185	175	4.29
24	1	1	0	185	275	6.74
25	1	0	0	185	53	1.30
26	1	0	0	124	150	3.68
27	1	1	0	124	356	8.72
28	1	2	0	124	224	5.49
29	1	2	0	77	193	4.73
30	1	1	0	77	106	2.60
31	1	0	0	77	300	7.35
32	1	0	0	46	84	2.06
33	1	1	0	46	277	6.79
34	1	2	0	46	189	4.63
35	1	2	0	15	227	5.56
36	1	1	0	15	93	2.28
37 center drilled	0	2	0	185	19	0.47
38	0	2	0	124	20	0.49
39	0	2	0	77	21	0.51
40	0	2	0	46	37	0.91
41	0	2	0	22	43	1.05
42	0	2	0	15	58	1.42
43	1	2	0	185	180	4.41
44	1	2	0	124	183	4.48
45	1	2	0	77	347	8.50
46	0	0	1	15	17	0.42
47	0	1	1	15	25	0.61
48	0	2	1	15	39	0.96
49	0	2	1	22	42	1.03
50	0	1	1	22	28	.69
51	0	0	1	22	20	.49
52	0	0	1	46	16	.39
53	0	1	1	46	20	.49
54	0	2	1	46	27	.66
55	0	2	1	77	21	.51
56	0	1	1	77	18	.44
57	0	0	1	77	13	.32
58	0	0	1	124	11	.27
59	0	1	1	124	12	.29
60	0	2	1	124	13	.32
61	0	2	1	185	13	.32
62	0	1	1	185	11	.27
63	0	0	1	185	8	.20
64	1	0	1	185	310	7.60
65	1	1	1	185	381	9.33
66	1	2	1	185	120	2.94
67	1	2	1	124	337	8.26
68	1	1	1	124	149	3.65
69	1	0	1	124	180	4.41
70	1	0	1	77	123	3.01
71	1	1	1	77	120	2.94
72	1	2	1	77	142	3.48
73	1	2	1	46	350	8.58

Table A-5 (Continued)

Drill #	a	b	c	sfm	# Holes	(cu in.) Material Removed
74	1	1	1	46	243	5.95
75	1	0	1	46	48	1.18
76	0	0	2	15	13	0.32
77	0	1	2	15	19	.47
78	0	2	2	15	30	.74
79	0	2	2	22	23	.56
80	0	1	2	22	21	.51
81	0	0	2	22	11	.27
82	0	0	2	46	10	.25
83	0	1	2	46	14	.34
84	0	2	2	46	16	.39
85	0	2	2	77	17	.42
86	0	1	2	77	16	.39
87	0	0	2	77	8	.20
88	0	0	2	124	7	.17
89	0	1	2	124	10	.25
90	0	2	2	124	13	.32
91	0	2	2	185	13	.32
92	0	1	2	185	10	.25
93	0	0	2	185	7	.17
94	1	0	2	15	20	.49
95	1	1	2	15	18	.44
96	1	2	2	15	34	.83
97	1	2	2	22	70	1.72
98	1	1	2	22	133	3.26
99	1	0	2	22	50	1.23
100	1	0	2	46	39	0.96
101	1	1	2	46	240	5.88
102	1	2	2	46	233	5.71
103	1	2	2	77	227	5.56
104	1	1	2	77	85	2.08
105	1	0	2	77	113	2.77
106	1	0	2	124	72	1.76
107	1	1	2	124	180	4.41
108	1	2	2	124	90	2.21
109	1	2	2	185	180	4.41
110	1	1	2	185	120	2.94
111	1	0	2	185	60	1.47
112 center drilled	1	2	0	15	42	1.03
113 ↓	1	2	0	22	43	1.05
114 ↓	1	2	0	46	50	1.23

Legend:

	0	1	2
a - Coating	Uncoated	Coated	
b - Feed Rate, in./rev	0.0015	0.003	0.006
c - Coolant	Dry	Lusol	Soluble Oil

Table A-6. TURNING DATA ON FIBERGLASS

Insert #	Experimental					Total Matl	
	Condition	A	B	C	D	E	Removed (cu in.)
1	1	0	0	0	0	0	123.8
2	a	1	0	0	0	0	47.6
3	b	0	1	0	0	0	118.6
4	ab	1	1	0	0	0	175.8
5	c	0	0	1	0	0	48.7
6	ac	1	0	1	0	0	13.5
7	bc	0	1	1	0	0	95.6
8	abc	1	1	1	0	0	294.8
9	d	0	0	0	1	0	40.3
10	ad	1	0	0	1	0	18.6
11	bd	0	1	0	1	0	103.7
12	abd	1	1	0	1	0	68.0
13	cd	0	0	1	1	0	112.1
14	acd	1	0	1	1	0	30.8
15	bcd	0	1	1	1	0	43.4
16	abcd	1	1	1	1	0	67.8
17	e	0	0	0	0	1	20.1
18	ae	1	0	0	0	1	21.8
19	be	0	1	0	0	1	122.5
20	abe	1	1	0	0	1	32.2
21	ce	0	0	1	0	1	41.0
22	ace	1	0	1	0	1	13.1
23	bce	0	1	1	0	1	80.7
24	abce	1	1	1	0	1	11.8
25	de	0	0	0	1	1	22.5
26	ade	1	0	0	1	1	24.5
27	bde	0	1	0	1	1	29.8
28	abde	1	1	0	1	1	114.0
29	cde	0	0	1	1	1	39.3
30	acde	1	0	1	1	1	171.8
31	bcde	0	1	1	1	1	27.5
32	abcde	1	1	1	1	1	170.5

Legend: A - Rake Angle      C - Feed Rate      E - Coating  
           0 - 5°                    0 - 0.01"/min      0 - Uncoated  
           1 - 10°                  1 - 0.02"/min      1 - Coated  
  
           B - Depth of Cut      D - Coolant  
           0 - 0.05"                0 - Dry  
           1 - 0.075"              1 - Soluble Oil  
    Coolant

Table A-7. END MILL TESTS ON FIBERGLASS

End Mill #	A	B	sfm	(cu in.) Material Removed
1	0	0	92	0.47
2	0	0	153	0.33
3	0	0	29	0.88
4	0	0	18	2.80
5	0	0	10	6.57
6	0	0	44	0.88
7	1	0	44	4.66
8	1	0	29	5.14
9	1	0	18	6.45
10	1	0	10	5.36
11	1	0	92	3.33
12	1	0	153	1.51
13	0	1	153	0.54
14	1	1	153	2.00
15	1	1	92	5.75
16	0	1	92	0.80
17	0	1	10	7.55
18	1	1	10	11.08
19	1	1	18	6.82
20	0	1	18	4.28
21	0	1	29	3.57
22	1	1	29	9.21
23	1	1	44	4.99
24	0	1	44	2.15

Legend: A - Coating      B - Coolant  
           0 - Uncoated      0 - Dry  
           1 - Coated        1 - Lusol Coolant



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TITANIUM DIBORIDE ELECTRODEPOSITED COATINGS -  
Jordan D. Kellner, William J. Croft, and  
Lawrence A. Shepard

Technical Report AMMRC TR 77-17, June 1977, 46 pp -  
illus-tables, D/A Project TT162102AH84,  
AMCMS Code 612105.H8400

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Abrasion resist coatings

Titanium boride